APPENDIX B

STANDARD OPERATING PROCEDURES

Bottle Selection and other Sampling Considerations When Sampling for Per- and Poly-Fluoroalkyl Substances (PFAS)

What type of samples does this guidance apply to?

This guidance applies to any sample taken for the analysis of per- and poly-fluoroalkyl substances (PFAS). This guidance is applicable to any liquid, soil, sediment, and tissue matrix.

Why do we need special sampling guidance for this?

PFAS are a class of manufactured compounds that are extensively used to make everyday items more resistant to stains, grease, and water. These chemicals have been used in a variety of industrial, commercial and consumer products. Some of these products could be present and/or used during a routine sampling event, such as plastic bags and bottles, waterproof clothing, detergents, and waterproof pens and paper. Because the EPA has established health advisory levels that are very low concentrations (70 parts per trillion) for two PFAS, perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS), the use of these products could possibly contaminate the samples during sample collection. This includes what is used to prepare the sampling site, what is used to collect the sample, what is used to clean the sampling equipment, what the sample is collected in, and how the sample is shipped. This guidance will provide steps to take to help you avoid these potential sources of contamination.

What type of bottle do I need to collect my sample in?

All samples to be analyzed for PFAS must be collected in a high density polyethylene (HDPE) container with an unlined plastic screw cap, except as stated below for drinking water samples by Method 537 for the method specified short list of PFAS. Polypropylene bottles may be used in this instance only.

Why can't we use the polypropylene bottles that are recommended in the drinking water method (EPA Method 537)?

EPA Method 537 is used for the analysis of a short list of PFAS. Polypropylene bottles can be used for this short list of analytes by this method. While some of these analytes do adsorb onto the polypropylene container, their adsorption is reversed by the rinsing of the sample bottle, which is required by the method. Other analytes have not been studied and other methods do not require the sample bottle to be rinsed. Therefore, as a precaution, use of HDPE bottles for all other PFAS sample collection is required.

What do we need to avoid using during sampling events?

Below is a general list of prohibited materials. Specific guidelines are determined based on project requirements.

PROHIBITED Materials and Equipment
Teflon®-containing materials, when possible, should be avoided (e.g., tubing, bailers, tape, and plumbing paste). In cases where Teflon®-containing materials are unavoidable, ensure adequate purging is performed prior to sampling (e.g., in-well pumps) and/or rinse blanks are collected prior to sampling.
LDPE or polypropylene containing materials (e.g., bags or containers used to transport samples)
Paper products such as waterproof field books, plastic clipboards, binders, spiral hard cover notebooks, sticky notes or glue materials
Markers
Chemical (blue) ice packs
Decontamination soaps containing fluoro-surfactants such as Decon 90
Water that is not verified to be "PFAS-free" to be used for trip and decontamination blanks and decontamination processes
Water resistant, waterproof, stain-treated clothing or shoes including Gore-Tex™ and Tyvek® materials

Bottle Selection and other Sampling Considerations When Sampling for Per- and Poly-Fluoroalkyl Substances (PFAS)

Is there anything else I should consider as a potential source of contamination?

Yes. There is some documentation that indicates that some personal care products, as well as food and drinks, may introduce additional ways your sample may get contaminated. Therefore, these additional precautions should be taken:

- Field personnel should not use cosmetics, moisturizers, hand cream, or other related products.
- Many manufactured sunblock and insect repellents contain PFAS and should not be used.
- No food or drink shall be brought on-site, with the exception of bottled water and hydration drinks.

What can we use for our sampling event instead?

Below is a general list of recommended materials. Specific guidelines are determined based on project requirements.

Recommended Materials and Equipment
HDPE and silicon Materials include: tubing, bailers, tape, plumbing paste
Acetate liners for direct push technologies
Nitrile gloves – change often
Loose paper with Masonite or aluminum clipboards
Pens
Bags of ice
Alconox® or Liquinox®
Laboratory supplied and verified "PFAS-free" water to be used for trip and decontamination blanks and decontamination processes
Cotton construction is recommended for field clothing and should be laundered a minimum of 6 times from time of purchase due to possible PFAS related treatments. Fabric softener must be avoided. Rain gear should be made from polyurethane and wax-coated materials.

Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods for Per- and Polyfluoroalkyl Substances (PFAS)

1 Introduction

PFAS contamination poses site characterization, sampling, and analytical challenges. PFAS have unique chemical and physical properties and they often occur in complex mixtures that can change over time. At environmental investigation sites, very low concentrations of several different PFAS must be sampled and analyzed. Many materials used in the course of environmental investigation can potentially contain PFAS. There is limited published research or guidance on how certain materials used by field staff affect sample results.

ITRC has developed a series of fact sheets that summarize the latest science and emerging technologies regarding PFAS. This fact sheet describes methods for evaluating PFAS in the environment, including:

- site characterization considerations
- sampling precautions
- laboratory analytical methods

USEPA has compiled an online resource for PFAS that includes topics such as policy and guidance, chemistry and behavior, occurrence, toxicology, site characterization, and remediation technologies (USEPA 2017h). The National Groundwater Association (NGWA) has also published a resource on PFAS that includes information about sampling and analytical methods (NGWA 2017).

2 Site Characterization Considerations

The purpose of site characterization is to understand the sources of contamination, site-specific contaminant fate and transport, and potential exposures and risks posed by a site. The site characterization techniques and study principles for PFAS-contaminated sites are generally the same as for any other site contaminated by hazardous substances. General site investigation principles and techniques will not be covered in this fact sheet, as these are well described in many existing guidance documents (for example, ASTM International 2011, 2013a, 2013b, 2014a, 2014b; Intergovernmental Data Quality Task Force (IDQTF) 2005; USEPA 1987, 1988a, 2000a, 2006c, 2013a, 2016i).

The unique chemical characteristics, uses, and transport mechanisms of PFAS should be accounted for when characterizing a contaminated site. PFAS sources (including ambient sources) pose many challenges, including their frequent occurrence as mixtures, the role of precursors, and the persistence and mobility of PFAS relative to other environmental contaminants.

2.1 Sources and Site Identification

The *Environmental Fate and Transport* fact sheet contains conceptual site models, including descriptions and figures, for four different common source scenarios. Phase 1 site characterization investigations (ASTM 2013c) may miss the potential for PFAS contamination at a site because these chemicals historically were not considered hazardous. Comparing timelines of site history (for example, processes, layout, chemical use, and release history) with the timeline of PFAS use and with existing drinking water data (for example, the UCMR3 data [USEPA 2017f]) can be helpful in determining source identification. A solid understanding of historical uses and the past presence of PFAS is critical to identifying PFAS that may have been released at a site. See the *History and Use* fact sheet for more information.

Another challenge is that commercial products and industrial releases may consist of complex PFAS mixtures that change over time through fate and transport mechanisms and may include unidentified PFAS. Changes in manufacturing practices as well as formula modifications also complicate the source identification. When characterizing source areas, there is often a focus on only perfluoroalkyl acids (PFAAs), particularly perfluorooctane sulfonate (PFOS) and perfluorooctanoate (PFOA), which are the current chemicals of concern. These and other chemicals of concern were often released as part of original PFAS mixtures, but also may be transformation products of PFAA precursors. The focus on PFAAs means that significant portions of the total PFAS contamination might be missed, leading to underestimates of plume life expectancy for groundwater and mass flux as well as PFAS contaminant mass.

The variation in mixtures of PFAS, associated with different processes and products, may provide signatures that help identify source areas and distinguish between multiple sources. However, careful analysis is needed to distinguish between signatures associated with differing sources and those due to environmental partitioning or multiple releases over time.

Knowledge of PFAS fate, transport, and mode of release is essential to placing sampling locations. Some PFAS released at aqueous film-forming foam (AFFF) training or application sites or by industrial air emissions may result in large, diffuse areas of soil contamination (rather than point sources) that act as sources of groundwater contamination. Air emissions

from industries using PFAS may result in releases to soil and surface water, with subsequent infiltration to groundwater (Davis et al. 2007; Shin et al. 2011).

2.2 Development of Initial Conceptual Site Model (CSM)

Conceptual site models for four different common source scenarios are included in the *Environmental Fate and Transport* fact sheet. These may be useful in developing a site-specific CSM. The CSM should include sources, site history, transport and exposure pathways, and receptor identification for a specific site. Any information pertaining to potential off-site PFAS contributors, such as landfills, wastewater treatment facilities, industrial sites, fire training areas and other sources, should be considered when determining possible secondary sources of PFAS.

2.2.1 Atmospheric, Geologic, and Hydrogeologic Framework

As with all contaminated sites, characterization relies upon an adequate understanding of the geology and hydrogeology of the site. Several PFAS, including the PFAAs of current regulatory concern, are relatively mobile in groundwater. Studies have reported both biotic and abiotic transformations of some polyfluorinated substances, referred to as precursors, which may form PFAAs. However, there is no evidence that PFAAs degrade or otherwise transform under ambient environmental conditions. Thus, PFAS plumes in groundwater may travel for several miles from the original source. At sites with highly permeable, low-organic matter soils, PFAS plumes can be extensive.

Partitioning behavior of perfluorocarboxylates (PFCAs) and perfluorosulfonates (PFSAs) has been studied more than that of other PFAS. PFCAs and PFSAs are organic anions at all environmentally relevant pH values and tend to be mobile in groundwater (Xiao et al. 2015). However, these compounds, especially those with longer carbon chains, often associate with the organic carbon fraction of soil or sediment (Higgins and Luthy 2006; Guelfo and Higgins 2013) when present in the saturated zone. See the *Environmental Fate and Transport* fact sheet for more information.

At sites where PFAS are detected in surface water, the CSM should address the potential for PFAS transport by surface water and infiltration of the PFAS to groundwater in areas downstream of the site. Some PFAS are highly soluble and resistant to breakdown in the environment, which means they may be transported significant distances in surface water (Awad et al. 2011; Kwadijk, Kotterman, and Koelmans 2014). In Minnesota, PFAS-contaminated surface water moving through a natural and manmade drainage system was found to have infiltrated to groundwater in multiple locations (losing streams, lakes, ditches, and stormwater ponds) creating large, discreet areas of groundwater contamination several miles from the original source areas (ATSDR 2008; MDH 2017).

A thorough understanding of the geology and hydrogeology of a site (including groundwater-surface water interactions and air-surface water interactions) can make selection of sampling locations more efficient and reduce the number of required samples. Without careful preparation, multiple, and sometimes redundant, field efforts can make site characterization costly.

2.2.2 Investigation Strategies

Many PFAS sites consist of releases that occurred decades before PFAS were regulated. As a result, contaminant plumes have had years to develop, and in some cases, stabilize. Therefore, site characterization should not necessarily proceed the same way as for newer sites with more recent releases. At these sites, sampling begins near the source area and steps outward to determine extent. For PFAS releases, however, contamination may have occurred in areas upgradient of drinking water sources, thus drinking water supply sampling should be a top priority to ensure that human receptors are protected. Data from private drinking water supply wells may be useful in determining the extent of contaminant plumes, if the well construction and characteristics information are available.

After evaluating drinking water, soils should be characterized to determine the three-dimensional extent of soil and groundwater contamination. Soil and groundwater sampling locations should be informed by fate and transport characteristics of the site type and source (see *Environmental Fate and Transport* fact sheet). Tools for determining the extent of established plumes may include transect surveys using direct push technology, followed by installation of monitoring wells, or other appropriate techniques such as high-resolution site characterization (USEPA 2016i). Potential secondary sources should be identified, for example, from irrigation or biosolids application, and other anthropogenic factors affecting fate and transport of PFAS-contaminated media.

Certain PFAS are present in ambient air, and may be elevated near sources such as landfills, WWTFs, fire training facilities, and manufacturing plants. Typical air sampling methods for PFAS include either glass fiber or quartz fiber filters and a sorbent material such as polymeric resin or polyurethane foam to collect both the particle and gas phases. Most

methodologies in the literature collect the particle phase and then the gas phase; however, some studies developed a method to collect the gas phase first followed by the particle phase in efforts to not overestimate the particle phase concentration (Barber et al. 2007; Jahnke 2007b, 2009; Ahrens et al. 2011a, 2012).

2.2.3 Risk Assessment

Site-specific risk assessment is informed by data and information iteratively collected in the site characterization. Of the many PFAS that may be found at contaminated sites, the toxicity of PFOA and PFOS has been studied the most thoroughly. A substantial database of toxicity information is also available for some other PFAS including PFBA, PFBS, PFHxA, PFNA, and GenX, while there is limited publicly available information on toxicity of other PFAS that may be present at PFAS-contaminated sites. USEPA has established a Health Advisory for protection from a lifetime exposure to PFOA and PFOS from drinking water of 70 ppt for each compound individually, or the total of both. While many states use these USEPA Health Advisories as guidance for PFOA and PFOS, several states have developed more stringent levels for these compounds; some states have also developed standards or guidance for other PFAS of local concern (see the *Regulations, Guidance, and Advisories* fact sheet). Given that PFAS typically occur in complex mixtures, and human and environmental receptors are exposed to some PFAS-forming complex mixtures, evaluating the true risks at a site can be particularly challenging. In the absence of risk-based values for some of the PFAS that are detected and because additional PFAS not detected by the analytical method may be present, the investigation team should identify data gaps and communicate the impact that these gaps have on risk analyses. Data gaps and scientific uncertainty must be documented so that as site cleanup progresses and more information becomes available, the project team can reassess potential risks from the site and better communicate to the public how site decisions are made.

2.2.3.1 Human Receptors

The presence of PFAS in the environment and consumer product has resulted in detectable levels (most frequently PFOA, PFNA, PFOS and PFHxS) in the blood serum of most of the U.S. population (CDC 2017b). The total body burden of these PFAS results from exposure to the PFAS themselves and formation from precursors through metabolism in the body (Olsen et al. 2017; D'eon and Mabury 2011). Blood serum levels of these PFAS in the general population have generally decreased over time (CDC 2017a). Risk assessment of PFAS exposure for humans near contaminated sites must include both exposures prevalent in the general population, such as from the food supply and consumer products, and exposures from the contaminated site, such as drinking water, house dust, ambient air, and locally caught fish. Exposures from even relatively low levels (for example, below 70 ng/L) of long-chain PFAS in drinking water are much higher than total exposures in the general population not impacted by a contaminated site (Bartell 2017).

The tendency of some PFAS to bioaccumulate (ATSDR 2015a) is also a critical component in evaluating potential health effects; food chain routes of exposure should be considered. For example, PFOS and longer-chain perfluorinated sulfonates, and PFNA and longer-chain perfluorinated carboxylates, are known to bioaccumulate in fish, including in species used for food (Conder et al. 2008). Also, as a result of chronic ingestion of water and exposure to other materials containing PFAS, women may carry PFAS in their blood and breast milk. These PFAS are transferred to their baby during pregnancy and through breast feeding. Serum levels of long-chain PFAS rapidly increase in breast fed infants due to the PFAS levels present in breast milk and the higher fluid consumption rates of infants (Mogensen et al. 2015; Winkens et al. 2017; Fromme et al. 2010; Verner et al. 2016a, b).

2.2.3.2 Ecological Receptors

PFAS present a potential hazard to wildlife by direct and dietary exposure on both individual and population levels (Environment Canada 2006, 2012). Numerous studies have shown PFAAs, particularly PFSAs, are globally present in wildlife and may bioaccumulate in birds, fish, and mammals (including livestock); other animal classes are less studied (Houde et al. 2011; Lupton et al. 2014; OECD 2013). Biomagnification (in which concentrations increase with increasing trophic level) appears to be more complicated, occurring in some food webs but not others (Franklin 2016; Fang et al. 2014). Effects of PFAS exposure on wildlife vary widely by species and PFAS compound. Ecological toxicity information for many PFAS compounds is currently unavailable, while for others, data is limited and still evolving. Therefore, as site characterization activities for PFAS occur, the current state of the science should be reviewed before calculating ecological risk. More information is included in the *Environmental Fate and Transport* fact sheet.

3 Sampling

Sampling conducted to determine PFAS concentrations in water, soil, sediment, air, biota and other sources is similar to that for other chemical compounds, but with several additional specific considerations and protocols. If regulatory procedures, methods, or guidelines are inconsistent with the needs of a PFAS sampling program, then the governing

agency should be contacted directly to determine an alternate approach or if an exception can be made. Other considerations for PFAS sampling include low laboratory detection limits, state and federal screening levels, and in some cases, cleanup criteria and potential for background concentrations of PFAS in the environment.

3.1 Equipment and Supplies

Many materials used in the course of environmental investigation can potentially contain PFAS. There is limited published research or guidance on how certain materials used by field staff affect sample results. Therefore, a conservative approach is recommended to exclude materials known to contain PFAS. Obtain and review all Safety Data Sheets (SDSs) before considering materials for use during PFAS sampling. Materials to avoid include:

- Teflon, polytetrafluoroethylene (PTFE)
- waterproof coatings containing PFAS
- food containers
- anything with fluoro in the name
- fluorinated ethylene propylene (FEP)
- ethylene tetrafluoroethylene (ETFE)
- low density polyethylene (LDPE), polyvinylidene fluoride (PVDF)

Many waterproof coatings contain PFAS, such as Gore-tex treated PPE or most waterproof papers, but some products are waterproofed with acceptable materials such as polyurethane, rubber, or PVC. Individual product specifications should be examined closely. In the case of Tyvek PPE, plain Tyvek does not contain PFAS while coated Tyvek does. In addition, materials incidentally transported to sites may contain PFAS. For example, fast food wrappers may contain PFAS. Due to the ubiquitous nature of PFAS, sampling crews must review all materials used to avoid contamination. Collection of quality assurance and quality control (QA/QC) samples is a useful tool to assess field contamination.

Two guidance documents identify materials and equipment that can be used in PFAS-focused investigations, as well as materials that should be avoided because they are known or suspected to be potential sources of PFAS:

- *Bottle Selection and other Sampling Considerations When Sampling for Per-and Poly-Fluoroalkyl Substances (PFAS)* (USDOD EDQW 2017b)
- *Interim Guideline on the Assessment and Management of Perfluoroalkyl and Polyfluoralkyl Substances (PFAS), Contaminated Sites Guidelines*, (Government of Western Australia, Department of Environment Regulation 2016)

Sometimes it is impossible to eliminate materials that affect PFAS results in samples. For example, these materials might be needed at sites where hazards warrant the use of specific personal protective equipment (PPE), where PFAS are the secondary or co-contaminant and the primary contaminant requires specific materials for proper sampling, or where the opportunity to collect a sample occurs before a proper sampling program is developed. When PFAS-containing equipment and supplies cannot be eliminated, increasing the equipment rinse blank samples will more thoroughly document the PFAS concentrations. In these situations, a thorough QA/QC program becomes even more important.

Not all PFAS are hydrophilic, and some are volatile. As a result, these chemicals may sorb to sampling equipment and supplies or be lost from samples during sample collection. Preliminary data suggest that sorption may occur quickly. Additionally, volatile losses have not yet been characterized. Until they are better quantified, sampling efforts should consider whether these losses would affect project objectives and adjust accordingly.

3.2 Bottle Selection and Sample Amount

Containers should be specified in the analytical method, provided by the laboratory selected to perform the analyses, and should be certified by the laboratory to be PFAS-free. The term *PFAS-free* is a method or project-defined concentration level (for example, < 1/2 the limit of quantitation for the specific compound of interest). USEPA Method 537, Version 1.1 (September 2009) requires the use of 250 mL polypropylene containers and caps/lids for drinking water sampling (Shoemaker, Grimmett, and Boutin 2009). Currently, USEPA has not issued guidance or analytical methods for any sample media other than drinking water. Depending on the analytical method used or program (for example state or DOD) requirements, polypropylene or high-density polyethylene (HDPE) bottles with unlined plastic caps are typically used (USDOD EDQW 2017b).

Best practices in sample preparation must be used when selecting the size, volume, and representativeness of samples. To minimize effects from analyte sorption on sample containers, the laboratory must analyze the entire sample, including the sample container rinsate. The project screening or applicable regulatory levels, and the expected or potential concentration of the analytes, are also relevant. If the sample is known to contain high concentrations of PFAS (for example, AFFF formulations), loss is negligible and therefore the entire sample does not need to be used.

Because the concentration level of PFAS in aqueous samples determines whether the whole sample or an aliquot is used in the laboratory preparation, the sampler should collect an additional volume of each sample in a separate container. Then, the laboratory can screen the extra sample for high concentrations without affecting the final sample result. For soil or sediment, obtaining a representative subsample in the laboratory is critical, so the entire sample should be homogenized in the laboratory prior to subsampling. Coordinating with the laboratory is crucial to determine the appropriate sample container volumes for environmental media other than drinking water.

3.3 Sample Preservation, Shipping, Storage, and Hold Times

USEPA Method 537, Version 1.1 contains specific requirements for drinking water sample preservation, shipping, storage, and holding times (Shoemaker, Grimmett, and Boutin 2009). Currently, there is no USEPA guidance or requirement for other sample media. The chemical preservation required by Method 537, Trizma, is added for buffering and free chlorine removal and applicable to DW samples only. Until additional information is available, the thermal preservation, shipping, storage, and holding times contained in USEPA Method 537, Version 1.1 should be used for all other sample media except biota. For biota samples (for example, vegetation, fish), the samples should be frozen to limit microbial growth until sample preparation is performed at the laboratory. Microbial growth may result in PFAAs values biased high due to biodegradation of precursor compounds; however, these effects have not been well studied.

3.4 Decontamination Procedures

Field sampling equipment, including oil/water interface meters, water level indicators, and other nondedicated equipment used at each sample location, require cleaning between use. The SDSs of detergents or soaps used in decontamination procedures should be reviewed to ensure fluoro-surfactants are not listed as ingredients. Use laboratory-certified PFAS-free water for the final rinse during decontamination of sampling equipment. Decontaminate larger equipment (for example, drill rigs and large downhole drilling and sampling equipment) with potable water using a high-pressure washer or steam. To the extent practical, rinse parts of equipment coming in direct contact with samples with PFAS-free water. Heavy equipment is best cleaned within a decontamination facility or other means of containment (for example, a bermed, lined pad and sump, or a portable, self-contained decontamination booth). Potable water sources should be analyzed in advance for PFAS. Wherever possible, rinse equipment with PFAS-free water immediately before use.

3.5 Field QC

Field quality control (QC) samples are a means of assessing quality from the point of collection. Such QC samples include, but are not limited to, field reagent blanks, equipment rinse blanks, and sample duplicates. USEPA Method 537, Version 1.1 contains specific requirements for the QC samples that must accompany drinking water samples. Collection and analysis of QC samples are important for PFAS analyses because of very low detection limits and widespread commercial use (historical and current) of PFAS containing products.

3.6 Sampling Precautions

Standard sampling procedures can be used at most PFAS sites. However, there may be some exceptions and additional considerations related to PFAS behavior, and issues associated with potential use of PFAS-containing or adsorbing sampling equipment and supplies.

3.6.1 Groundwater

The most inert material (for example, stainless steel, silicone, and HDPE), with respect to known or anticipated contaminants in wells should be used whenever possible. Dedicated sampling equipment installed in existing wells prior to investigation should be thoroughly checked to ensure that the equipment is PFAS-free. For long-term investigations, samples may be collected in duplicate with and without existing dedicated equipment. If PFAS analyses show that the equipment does not affect results, the equipment may be kept and used long term. This determination depends on project-specific requirements, however, and should only be used by a project team with full disclosure to all stakeholders.

3.6.2 Surface Water

To avoid cross-contamination from sampling materials to sample media, the outside of all capped sample containers should be rinsed multiple times with the surface water being sampled before filling the containers. When site conditions require, remote sampling into sample containers can be accomplished by clamping the container onto the end of a clean extension rod. The extension rod must be made of PFAS-free material and have been decontaminated. Within the context of sample collection objectives, the sample location in the water column should consider the potential stratification of PFAS in solution and their tendency to accumulate at the air/water interface. For more information on stratification, see the *Environmental Fate and Transport* fact sheet.

3.6.3 Porewater

Peristaltic pumps with silicone and HDPE tubing are typically used for porewater sample collection, along with push point samplers, porewater observation devices (PODs), or drive point piezometers. Push point samples and drive point piezometers are made of stainless steel, while PODs consist of slotted PVC pipe and silicone tubing. These samplers should be dedicated and not reused across a site or multiple sites.

3.6.4 Soil/Sediment

Most core and grab sampling devices are constructed of stainless steel. Some core samplers include an HDPE sleeve inserted in the core barrel to retain the sample. PPE such as waders and personal flotation devices may be required. Ensure that materials that contact the media to be sampled do not have water-resistant coatings which contain PFAS.

3.6.5 Fish

The species of fish collected, as well as the portion of fish sampled (whole versus fillet), depends on the project goals (for example, ecological risk or human health). Studies have shown the majority of the PFAS in fish are stored in the organs, not the flesh (Martin et al. 2004; Yamada et al. 2014). Communicating project objectives to the laboratory is important prior to field work in order to determine the necessary quantity and quality of tissue, fish handling requirements, laboratory sample preparation (including single fish or composite fish samples, and whole or fillet preparation), and packing and shipping requirements.

3.6.6 Potential high concentration samples

The CSM or previous sampling may indicate areas of high concentrations of PFAS for which single-use, disposable equipment is recommended. If single-use is not possible, take additional precautions such as implementing a greater frequency of decontamination blanks and not reusing equipment to sample potentially low PFAS concentration samples. High concentration samples should be segregated during shipping to the laboratory.

Some projects may require the analysis of AFFF product that has been used at the site. All AFFF product samples must be considered high concentration samples. These samples should be segregated from other samples during sampling and shipping to avoid cross contamination. Samples that may contain high concentrations of PFAS should be clearly identified on the *Sample Chain of Custody* that is shipped with the samples. Field test kits are available for PFAS but have not been fully evaluated. While these kits cannot achieve low detection limits, they could be helpful in screening for potential high concentrations of PFAS in the field.

4 Quantitative Analysis

USEPA Method 537, Version 1.1 contains specific requirements for sample preparation and analysis of drinking water samples. Currently, there are no USEPA methods for the preparation and analysis of other sample media. However, other published methods may apply:

- ISO Method 25101 (ISO 2009)
- ASTM D7979 (ASTM 2017b)
- ASTM D7968 (ASTM 2017a)

To evaluate the laboratory's ability to meet the needs of a project, the laboratory's analytical procedure should be reviewed as part of the laboratory selection process. In addition, performance data such as concentrations observed in lab blanks and matrix spike recovery are necessary.

4.1 Sample Preparation

The sample preparation procedure should be specified in the sample analysis procedure and should be included as part of the sample and analysis plan (SAP) or quality assurance project plan (QAPP). This procedure should demonstrate that extreme care is taken to prevent sample contamination during preparation and extraction. All supplies must be checked and confirmed as PFAS-free prior to sample preparation. Intermittent contamination can occur due to vendor supply or manufacturing changes; therefore, each lot of supplies should be verified and documented prior to use.

Because sample preparation may vary in different analytical procedures, the laboratory should document its preparation process for the samples. A critical step in the laboratory's preparation process is ensuring a representative sample or subsample is used for analysis. For all media, sample transfers should be minimized. Sample filtration to eliminate solid particulate from aqueous samples is not recommended because PFAS losses can occur due to adsorption of PFAS onto filters.

The entire aqueous sample received should be prepared and the sample container appropriately rinsed. Aqueous samples that are prepared using the whole sample must be extracted using SPE. The exception to this practice is samples containing high concentrations of PFAS, because each type of solid phase extraction cartridge has a defined capacity to retain PFAS analytes. Exceeding this capacity results in a low bias in PFAS results. In these instances, to prevent this bias, samples can be prepared using serial dilution techniques or analyzed using direct injection (for example, ASTM D7979). Most laboratories screen samples using a small volume sample to determine if it contains PFAS at concentrations too high for SPE sample preparation and analysis. For solid samples, the laboratory homogenizes the sample before subsampling and extraction.

To account for biases resulting from preparation steps, internal standards should be added to all samples (preferably extracted internal standards that are isotopically-labeled analogs of each analyte, if commercially available). The addition of internal standards to the sample should be clearly documented. Internal standards should be added to the sample at different steps in the process, depending on the sample preparation process used. Internal standards should also be added to whole field samples in the field container (SPE extraction samples) after subsampling, prior to addition of extraction solvent for soil or sediment samples, and after final dilution for serial dilution prepared samples (USDOD 2017a).

Depending on the analytical method used, cleanup procedures (for example, graphitized carbon) may be used on samples when matrix interferences (for example, bile salts and gasoline range organics) could be present. ENVI-Carb cleanup removes cholic acids, a known interference in fish tissue sample. The procedure should clearly state what type of cleanup process is used and in what instances.

The analytical procedure should describe what batch QC samples are prepared with each media type. Batch QC samples might include method blank (MB), laboratory control sample (LCS), laboratory control sample duplicate (LCSD), sample duplicate (SD), matrix spike (MS), and matrix spike duplicate (MSD). Additional QC may also be included. For samples with high concentrations of PFAS, in addition to an MS and an MSD, an LCSD and an SD may be warranted. The SD should be prepared using a different aliquot from the same sample bottle to create a second set of serial dilutions. Review of the laboratory's procedure should ensure that the laboratory is capable of using the batch QC needed for the project, including meeting the project's QC acceptance criteria.

4.2 Sample Analysis

Currently, the analytical detection method of choice for PFAS analysis is liquid chromatography-mass spectrometry-mass spectrometry (LC/MS/MS), which is especially suited for analysis of ionic compounds, such as the PFSAs and PFCAs. Gas chromatography-mass spectrometry (GC/MS) can also be used for PFAS analysis, specifically the neutral and nonionic analytes, such as the fluorotelomer alcohols (FTOHs), perfluoroalkane sulfonamides, and perfluoroalkane sulfonamido ethanols. Currently, LC/MS/MS analysis of PFAS is widely available, whereas GC/MS analysis has limited commercial availability.

LC/MS/MS methods developed by laboratories may be based on USEPA Method 537, Version 1.1. The USEPA method does not contain steps to alleviate matrix interference issues potentially found in other sample media and does not contain steps to prepare solid sample media. Methods for other sample media may include extraction or sample preparation procedures for other matrices, use of isotope dilution, the addition of other PFAS analytes, and confirmation using confirmatory ions and ion ratios. Because these modifications are not standardized, analytical methods can result in greatly varied data, precision, and accuracy. Laboratories should provide performance data for the relevant media

for each project. The USDOD EDQW has attempted to standardize many of these modifications through requirements contained in the USDOD Environmental Laboratory Accreditation Program (USDOD ELAP) document, the DOD *Quality Systems Manual for Environmental Laboratories* (DOD QSM), Version 5.1, Appendix B, Table B-15 (USDOD 2017a).

Certified analytical standards are available from several manufacturers. Products may have variable purity and isomer profiles, which may compromise the accuracy, precision, and reproducibility of data. Only certified standards of the highest purity available, for example, American Chemical Society grade, can be used for accurate quantitation. Standards containing linear and branched isomers are not commercially available for all applicable analytes. Currently, such standards are only available for PFOS and perfluorohexane sulfonic acid (PFHxS). Technical grades which contain branched and linear isomers are available for other PFAS, but these standards do not have the accuracy needed for quantitation purposes. These standards may, however, be qualitatively useful for verifying which peaks represent the branched isomers. Methods should specify the isomers quantified as well as the isomers included in standards used for quantitation purposes.

Isotope dilution is a quantitation technique that considers sample matrix effects on each individual PFAS quantitation in the most precise manner possible. This technique quantifies analytes of interest against the isotopically labeled analogs of the analytes, which are added to the sample prior to and after sample preparation. Addition prior to preparation helps account for loss of analyte during the preparation process, while addition after preparation to an aliquot of the sample extract accounts for the bias associated with the instrumentation. Methods using isotope dilution should include isotope recovery for each sample and analyte in data reports. Isotope analog recoveries should be reported, and minimum/maximum isotope recoveries may be required by specific analytical procedures. Low isotope recovery may indicate that quantitation was inadequate; the data are then reported as estimated values.

Mass calibration should occur at the frequency recommended by the instrument manufacturer and as needed based on QC indicators, such as calibration verifications. The instrument blanks, calibration curve, and initial and continual calibration verification requirements should be consistent with those published for other LC/MS/MS methods. The lowest calibration point should be a concentration at or below the limit of quantitation. A standard at the limit of quantitation concentration should be analyzed with each analytical batch to document the instrument's ability to accurately quantitate down to that concentration. Instrument blanks are critical in determining if the instrument is potentially affecting PFAS concentrations in samples.

Quantification by LC/MS/MS may be accomplished using a variety of techniques. For relatively simple matrices such as drinking water, Method 537 quantifies analytes by comparing the product ion of one precursor ion and retention time in samples to calibration standards. For more complex matrices, additional product ions and their ion ratios can be used to distinguish analytes from matrix interference. In an MS/MS system, an analyte can be fractured into more than one ion. By monitoring the area of each ion and comparing the ratio of those area counts, a more definitive identification can be made. This identification allows the analyst to distinguish true target analytes from false positives. This more detailed quantification is not required for drinking water matrices, but it is useful for more complex matrices.

As part of the laboratory selection process, the laboratory's analytical procedure should be evaluated to ensure these parameters are addressed in the documentation provided. In addition, the acceptance criteria for all the analytical QC elements should be evaluated to ensure that they are set at levels that meet the project's measurement quality objectives (MQOs). For DOD projects, these criteria can be found in the DOD QSM, Version 5.1, Appendix B, Table B-15 (USDOD 2017a).

4.3 Data Evaluation

Data evaluation is a critical step in any project; however, it becomes even more important when nonstandard methods are used, such as for PFAS. Without a standard method for media other than drinking water, laboratories' methods may vary greatly in their precision and accuracy. Over time, these methods become optimized based on new knowledge about sampling and analytical biases. Advances in instrumentation and analytical supplies (such as standards availability and improved analytical columns) often occur as well because of commercial demand. As a result, the precision and accuracy of the data generated by laboratories can change significantly over time, making it difficult to compare data generated over an extended time period. Thus, data evaluation should be performed using the most current knowledge on the state of science of PFAS.

Precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS) parameters should be assessed because they guide data evaluation (field collection and laboratory information). Data are reviewed in a

systematic way by looking at the results of each QC indicator of the PARCCS parameters (for example, spike recoveries and method blanks) to obtain an understanding of the overall quality of the data. The most important goal of data evaluation is to ensure that any limitations to the PFAS data generated are understood, which establishes confidence that the data meet site-specific needs. More information is available in the IDQTF (2005) and USEPA (2000a) Quality Assurance Project Plan documents.

5 Qualitative Analysis

Several methods employing indirect measurement have been developed that more comprehensively assess the range of PFAS contamination at a site. Two techniques are available to measure organofluorine (Dauchy et al. 2017; Willach, Brauch, and Lange 2016; Ritter et al. 2017):

- Adsorbable organic fluorine (AOF) paired with combustion ion chromatography (CIC) measure the combusted organofluorine content of a sample as fluoride on an IC.
- Proton induced gamma-ray emission (PIGE) spectroscopy measures elemental fluorine isolated on a thin surface.

Both techniques isolate organofluorine material on a sorptive material such as activated carbon or an anion exchange cartridge prior to measurement; neither technique is currently commercially available. A third technique, total oxidizable precursor assay (TOP assay or TOPA) converts PFAA precursor compounds to PFAAs through an oxidative digestion. The increase in PFAAs measured after the TOP assay, relative to before, is a conservative estimate of the total concentration of PFAA precursors present in a sample, because not all PFAS present will be subject to quantitation or reaction, and will remain as undetected PFAS. The PFAAs generated have perfluoroalkyl chain lengths equal to, or shorter than, the perfluoroalkyl chain lengths present in the precursors (Houtz et al. 2013; Houtz and Sedlak 2012; Weber et al. 2017; Dauchy et al. 2017). Finally, quantitative time of flight mass spectrometry (QTOF-MS) can be used to determine both the chemical formula and structure of unknown PFAS in a sample, but analytical standards are required for unequivocal structural identification.

Library research, preliminary identification of potential PFAS sources, and information gathered from patents can assist in the identification of PFAS using QTOF-MS (Newton et al. 2017; Moschet et al. 2017; Barzen-Hanson et al. 2017). These methods are not standardized through a published USEPA method and range in commercial availability. To date, these methods have not undergone multilaboratory validation. As a result, TOP assay, the most widely commercially available of the techniques, is typically accepted as a means of determining PFAS load on remediation substances to estimate the replacement cycle, but not for site characterization.

6 References and Acronyms

The references cited in this fact sheet, and the other ITRC PFAS fact sheets, are included in one combined list that is available on the ITRC web site. The combined acronyms list is also available on the ITRC web site.

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INTRODUCTION

The following Standard Operating Procedure (SOP) is to describe the procedures for collecting representative soil samples. Analysis of soil samples may determine whether concentrations of specific soil pollutants exceed established action levels, or if the concentrations of soil pollutants present a risk to public health, welfare, or the environment. This SOP is similar to SOP Number 1001.03 for collecting near surface soil samples with a hand auger.

PROCEDURE

Surface soil samples may be collected using a variety of methods and equipment. The methods and equipment used are dependent on the depth of the desired sample, the type of sample required (disturbed versus undisturbed), and the type of soil. Near-surface soils may be easily sampled using a spade, trowel, or hand scoop.

Sample Preservation

Cooling to 4°C ± 2°C, supplemented by a minimal holding time, is suggested.

Interferences and Potential Problems

There are two primary interferences or potential problems associated with soil sampling: cross-contamination of samples and improper sample collection. Cross-contamination problems can be eliminated or minimized through the use of dedicated (disposable) sampling equipment. If this is not possible or practical, then decontamination of sampling equipment is necessary. Improper sample collection can involve using contaminated equipment, disturbance of the matrix resulting in compaction of the sample, or inadequate homogenization of the samples where required, resulting in variable, non-representative results. Homogenization may also affect sample representativeness where the analytical requirements include volatile organic compounds.

Equipment or Apparatus

The equipment used for sampling may be selected from the following list, as appropriate:

- Tape measure
- Survey stakes or flags
- Stainless steel, plastic, or other appropriate homogenization bucket or bowl
- Ziploc plastic bags
- Logbook
- Labels
- Chain-of-custody forms and seals
- Coolers
- Ice
- Decontamination supplies and equipment
- Canvas or plastic sheet
- Spatulas/spades/shovels
- Scoops

- Plastic or stainless steel spoons
- Trowel

Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and what equipment and supplies are required.
2. Obtain necessary sampling and monitoring equipment from the list above.
3. Prepare schedules, and coordinate with staff, client, and regulatory agencies, if appropriate.
4. Perform a general site survey prior to site entry in accordance with the site-specific health and safety plan.
5. Decontaminate or preclean equipment, and ensure that it is in working order.
6. Use stakes, buoys, or flagging to identify and mark all sampling locations. Consider specific site factors, including extent and nature of contaminant, when selecting sample locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. All staked locations will be utility-cleared by the property owner or other responsible party prior to soil sampling.
7. Evaluate safety concerns associated with sampling that may require use of personal protective equipment and/or air monitoring.

Surface Soil Sample Collection

Collect samples from the near-surface soil with tools such as spades, shovels, and scoops. Surface material can be removed to the required depth with this equipment, then a stainless steel or plastic scoop can be used to collect the sample. The use of a flat, pointed mason trowel to cut a block of the desired soil can be helpful when undisturbed profiles are required. A stainless steel scoop, lab spoon, or plastic spoon will suffice in most other applications. Avoid the use of devices plated with chrome or other target analyte materials.

The following procedures should be followed when collecting surface soil samples:

1. Carefully remove the top layer of soil or debris to the desired sample depth with a pre-cleaned spade.
2. Using a pre-cleaned, stainless steel scoop, plastic spoon, or trowel, remove and discard a thin layer of soil from the area which came in contact with the spade.
3. If volatile organic analysis is to be performed, transfer a portion of the sample directly into an appropriate, labeled sample container(s) with a stainless steel lab spoon, plastic lab spoon, or equivalent and secure the cap(s) tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into an appropriate, labeled container(s) and secure the cap(s) tightly; or if composite samples are to be collected, place a sample from another sampling interval into the

homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled container(s) and secure the cap(s) tightly.

4. Fill hole created through sampling with unused material or other appropriate backfill material (sand).

5. Record applicable information into field log book or appropriate forms as documentation of sampling.

SOP	**1001.10**				
GROUP	Soil Sampling Procedures				
SUB-GROUP					
TITLE	Soil Compositing				
DATE	4/24/2013	**FILE**	Compositing Soil Sampling - Revised 1001-10	**PAGE**	1 of 2

INTRODUCTION

The following Standard Operating Procedure (SOP) describes the procedure for compositing soil samples. Soil samples are typically collected for laboratory analysis, and sometimes it is necessary to composite (mix together) samples from several locations for one combined analysis at the laboratory. This soil sampling procedure is closely related to SOP Nos. 1001.01, 1001.03, and 1001.10 regarding soil sampling procedures. This procedure serves as an alternative method of sample preparation prior to placing the samples in containers, as described in the other named SOPs.

PROCEDURE

Equipment

Equipment that may be used as part of the soil compositing procedure is identified under SOP Nos. 1001.01 and 1001.03 where soil sampling methods are described. Specific equipment typically used during the compositing process after discrete samples are collected includes:

- Mixing bowls or buckets
- Scoops, spatulas, and knives
- Sample containers
- Personal protection clothing
- Plastic Sheeting
- Decontamination equipment and supplies

Method

The procedure to be used to physically collect soil samples are described in SOP Nos. 1001.01 and 1001.03. Reference should be made to these SOPs for specific sampling equipment, procedures, and other general guidelines. As soil samples are collected, the site-specific Sampling and Analysis Plan may require compositing (mixing together) of two or more samples to create a single sample that will be sent to the laboratory for analysis. When this is the case, the following compositing procedure will generally be used:

- The soil will be collected in general accordance with SOP 1001.01 or 1001.03, with the exception that samples from discrete locations will generally not be immediately placed into sample containers and an additional preparation step (i.e., compositing) will be performed.

- As they are collected, soil samples selected for compositing will be staged in a clean mixing bowl or mixing bucket until each sample to be included in the composite sample is obtained. Depending on site requirements and analytical procedures to be requested, it may be necessary to temporarily stage individual discrete-location samples within clean sample jars, aluminum foil, or other appropriate materials for the project. The method for sample staging should be specified in the site-specific sampling and analysis plan.

- For composite samples that will be analyzed for volatile organic compounds, an equal portion of soil will be removed directly from each discrete-location sample and placed into a final sample jar without homogenizing the soil.

- For analyses other than volatile organics, equal portions of soil will be removed from each discrete-location sample and placed in a clean mixing bowl. The equal portions of the samples will then be broken up and homogenized together using a scoop or spatula. Homogenization will generally continue until the discrete samples being combined are reasonably indistinguishable as individual samples in the soil mixture. However, it is recognized that homogenization can be difficult for highly plastic clays. In this case, equal amounts of the soil core of each clay sample will be cut into small, roughly cubical pieces using a stainless steel knife, and an equal numbers of pieces of each discrete sample will be placed into the bowl and homogenized to extent practical.

- The composited soil sample will be collected from the mixing bowl containing the individual homogenized samples after homogenization is performed. The composited sample will be collected using a stainless steel or disposable plastic scoop or similar tool. The sample will be placed in a clean sample container and then handled in accordance with soil sampling SOPs 1001.01 and 1001.03.

Variations on this procedure are allowable to accommodate different soil conditions and any site requirements specifically identified in the site-specific Sampling and Analysis Plan.

The number of discrete samples that may be composited into a single sample typically ranges from two to six. The number of discrete samples that may be composited for the project in question will be specified in the site-specific Sampling and Analysis Plan.

REFERENCES

SOP No. 1001.01 - Standard Operating Procedure, Surface Soil Sampling
SOP No. 1001.03 - Standard Operating Procedure, Shallow Subsurface and Near Surface Soil Sampling

INTRODUCTION

The following Standard Operating Procedure (SOP) is to describe the procedures for collecting representative surface water samples. Analysis of surface samples may determine whether concentrations of specific soil pollutants exceed established action levels, or if the concentrations of pollutants present a risk to public health, welfare, or the environment.

PROCEDURE

Surface water samples may be collected using a variety of methods and equipment. The methods and equipment used are usually dependent on the location of the body of water being sampled. Sampling can be performed by merely submerging the sample container, a weighted-bottle sampler with stopper, a bailer, or by pump assisted methods. Several types of pumps can be used for sampling depending on the objectives of sampling and the site conditions.

Sample Preservation

Samples are to be preserved in conformance with the site-specific Quality Assurance Project Plan, Sampling and Analysis Plan or work plan. In general these requirements include refrigeration to 4°C, addition of appropriate additives (HCl, H_2SO_4, NaOH) to adjust and fix pH, and a defined maximum holding time. If a site-specific plan is not available, the analytical laboratory should be consulted for the appropriate preservation procedures.

Interferences and Potential Problems

There are two primary interferences or potential problems associated with surface water sampling: cross-contamination of samples and improper sample collection. Cross-contamination problems can be eliminated or minimized through the use of dedicated sampling equipment. If this is not possible or practical, then decontamination of sampling equipment is necessary. Improper sample collection can involve using contaminated equipment, undue disturbance of the sample matrix, or improper sample location.

Equipment or Apparatus

- Ziploc plastic bags
- Logbook
- Labels
- Chain-of-custody forms and seals
- Coolers
- Ice
- Decontamination supplies and equipment
- Discharge tubing
- Sample containers
- Sampling devices

Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and which equipment and supplies are required.
2. Obtain necessary sampling and monitoring equipment.
3. Decontaminate or preclean equipment, and ensure that it is in working order.
4. Prepare schedules, and coordinate with staff, client, and regulatory agencies, if appropriate.
5. Perform a general site survey prior to site entry in accordance with the site-specific health and safety plan.

Surface Water Sampling

Samples from shallow depths can be readily collected by merely submerging the sample container. In flowing surface water bodies, the container's mouth should be positioned so that it faces upstream, while the sampling personnel stand downstream so as not to stir up sediment that could potentially contaminate the sample.

Collecting a representative sample from a larger body of surface water requires that samples be collected near the shore unless boats are feasible and permitted. If boats are used, the body of water should be cross sectioned, and samples should be collected at various depths across the body of water in accordance with the specified sampling plan. For this type of sampling, a weighted-bottle sampler is used to collect samples at a predetermined depth. The sampler consists of a glass bottle, a weighted sinker, a bottle stopper, and a line that is used to open the bottle and to lower and raise the sampler during sampling. The procedure for use is as follows:

- Assemble the weighted bottle sampler.
- Gently lower the sampler to the desired depth so as not to remove the stopper prematurely.
- Pull out the stopper with a sharp jerk of the sampler line.
- Allow the bottle to fill completely, as evidenced by the cessation of air bubbles.
- Raise the sampler and cap the bottle.
- Wipe the bottle clean. The sampling bottle can be also be used as the sample container for shipping.

Teflon bailers have also been used where feasible for collecting samples in deep bodies of water.

Another method of extending the reach of sampling efforts is the use of a small peristaltic pump. In this method the sample is drawn through heavy-wall Teflon tubing and pumped directly into the sample container. This system allows the operator to reach into the liquid body, sample from depth, or sweep the width of narrow streams.

The general sampling procedures are listed below:

1. Collect the sample using whichever technique, submerged bottle, bottle sampler with stopper, pump & tubing, or bailer.

2. The collected sample may be collected in the sample containers or may be transferred to the appropriate sample containers in order of the volatile organics first and inorganics last.

3. Label sample containers, place on ice in a cooler, remove, and decontaminate equipment as necessary.

REFERENCES

SOP 0110.01 Sample Nomenclature
SOP 1005.01 Field Duplicate Collection
SOP 1005.02 Rinse Blank Preparation
SOP 1005.03 Field Blank Preparation
SOP 1101.01 Sample Custody - Field
SOP 1102.01 Sample Shipping
SOP 1201.01 Sampling Equipment Decontamination
SOP 1501.01 Field Logbook

SOP	**1101.01**				
GROUP	Sampling Handling				
SUB-GROUP	Sample Custody				
TITLE	Sample Custody in the Field				
DATE	**11/19/2001**	**FILE**	1101-01.DOC	**PAGE**	1 of 4

INTRODUCTION

The following Standard Operating Procedure (SOP) presents procedures for maintaining sample chain of custody (COC) during activities where samples are collected.

PROCEDURE

Sample custody is defined as being under a person's custody if any of the following conditions exist:

- it is in their possession,
- it is in their view, after being in their possession,
- it was in their possession and they locked it up, or
- it is in a designated secure area.

A designated field sampler will be personally responsible for the care and custody of collected samples until they are transferred to another person or properly dispatched to the laboratory. To the extent practicable, as few people as possible will handle the samples.

Sample tags or labels will be completed and applied to the container of each sample. When the tags or labels are being completed, waterproof ink will be used. If waterproof ink is not used, the tags or labels will be covered by transparent waterproof tape. Sample containers may also be placed in Ziploc-type storage bags to help keep them clean in the cooler. Information typically included on the sample tags or labels will include the following:

- Project Code
- Station Number and Location
- Sample Identification Number
- Date and Time of Sample Collection
- Type of Laboratory Analysis Required
- Preservation Required, if applicable
- Collector's Signature
- Priority (optional)
- Other Remarks

Additonal information may include:

- Anticipated Range of Results (Low, Medium, or High)
- Sample Analysis Priority

A COC form will be completed each time a sample or group of samples is prepared for transfer to the laboratory. The form will repeat the information on each of the sample labels and will serve as documentation of handling during shipment. The minimum information requirements of the COC form are listed in Table 1101.01-A. An example COC form is shown in Figure 1101.01-A. The completed COC must be reviewed by the Field Team Leader or Site Manager prior to sample shipment. The COC form will remain each sample shipping container at all times, and another copy will be retained by the member of the sampling team who originally relinquished the samples or in a project file.

TABLE 1101.01-A CHAIN OF CUSTODY FORM

INFORMATION	COMPLETED BY	DESCRIPTION
COC	Laboratory	enter a unique number for each chain of custody form
SHIP TO	Field Team	enter the laboratory name and address
CARRIER	Field Team	enter the name of the transporter (e.g., FedEx) or handcarried
AIRBILL	Field Team	enter the airbill number or transporter tracking number (if applicable)
PROJECT NAME	Field Team	enter the project name
SAMPLER NAME	Field Team	enter the name of the person collecting the samples
SAMPLER SIGNATURE	Field Team	signature of the person collecting the samples
SEND RESULTS TO	Field Team	enter the name and address of the prime contractor
FIELD SAMPLE ID	Field Team	enter the unique identifying number given to the field sample (includes MS, MSD, field duplicate and field blanks)
DATE	Field Team	enter the year and date the sample was collected in the format M/D (e.g., 6/3)
TIME	Field Team	enter the time the sample was collected in 24 hour format (e.g., 0900)
MATRIX	Field Team	enter the sample matrix (e.g., water, soil)
PRESERVATIVE	Field Team	enter the preservative used (e.g., HNO3) or "none"
FILTERED/ UNFILTERED	Field Team	enter "F" if the sample was filtered or "U" if the sample was not filtered
CONTAINERS	Field Team	enter the number of containers associated with the sample
MS/MSD	Field Team or Laboratory	enter "X" if the sample is designated for the MS/MSD
ANALYSES REQUESTED	Field Team	enter the method name of the analysis requested (e.g., SW6010A)
COMMENTS	Field Team	enter comments
SAMPLE CONDITION UPON RECEIPT AT LABORATORY	Laboratory	enter any problems with the condition of any sample(s)
COOLER TEMPERATURE	Laboratory	enter the internal temperature of the cooler, in degrees C, upon opening
SPECIAL INSTRUCTIONS/COMMENTS	Laboratory	enter any special instructions or comments
RELEASED BY (SIG)	Field Team and Laboratory	enter the signature of the person releasing custody of the samples
COMPANY NAME	Field Team and Laboratory	enter the company name employing the person releasing/receiving custody
RECEIVED BY (SIG)	Field Team and Laboratory	enter the signature of the person receiving custody of the samples
DATE	Field Team and Laboratory	enter the date in the format M/D/YY (e.g., 6/3/96) when the samples were released/received
TIME	Field Team and Laboratory	enter the date in 24 hour format (e.g., 0900) when the samples were released/received

FIGURE 1101.01-A CHAIN OF CUSTODY FORM

SOP	**1102.01**				
GROUP	Sample Handling				
SUB-GROUP	Sample Shipping				
TITLE	Sample Shipping				
DATE	11/19/2001	**FILE**	1102-01.DOC	**PAGE**	1 of 1

INTRODUCTION

The following Standard Operating Procedure (SOP) presents the procedures for sample shipping that will be implemented during field work involving sampling activities.

TERMS

COC - Chain-of-Custody

PROCEDURE

Prior to shipping or transferring custody of samples, they will be packed according to D.O.T. requirements with sufficient ice to maintain an internal temperature of 4°C ± 2°C during transport to the laboratory. Samples relinquished to the participating laboratories will be subject to the following procedures for transfer of custody and shipment:

1. Samples will be accompanied by a COC record. When transferring possession of samples, the individuals relinquishing and receiving the samples will sign, date, and note the time of the sample transfer on the record. If sent by common carrier, a bill of lading or airbill should be used. Bill of lading and airbill receipts will be retained in the project file as part of the permanent documentation of sample shipping and transfer. This custody record documents transfer of sample custody from the sampler to another person or to the laboratory. The designated laboratory will accept custody in the field upon sample pick-up or at the laboratory if the samples are delivered via field personnel or a courier service.
2. Samples will be properly packed in approved shipping containers for laboratory pick-up by the appropriate laboratory for analysis, with separate, signed custody records enclosed in each sample box or cooler. Sample shipping containers will be padlocked or custody-sealed for transfer to the laboratory. The preferred procedure includes use of a custody seal wrapped across filament tape that is wrapped around the package at least twice. The custody seal will then be folded over and stuck to itself so that the only access to the package is by cutting the filament tape or breaking the seal to unwrap the tape. The seal will then be signed. The designated laboratory will accept custody of the samples upon receipt.
3. Whenever samples are split with state representatives or other parties, the COC record will be marked to indicate with whom the samples were split.
4. The field sampler will call the designated laboratory to inform them of sample shipment and verify sample receipt as necessary.

INTRODUCTION

The following Standard Operating Procedure (SOP) presents the methods used for minimizing the potential for cross-contamination, and provides general guidelines for sampling equipment decontamination procedures.

PROCEDURE

As part of the Health and Safety Plan (HASP), develop and set up a decontamination plan before any personnel or equipment enter the areas of potential exposure. The decontamination plan should include the following:

- The number, location, and layout of decontamination stations
- Which decontamination apparatus is needed
- The appropriate decontamination methods
- Methods for disposal of contaminated clothing, apparatus, and solutions

Decontamination Methods

Personnel, samples, and equipment leaving the contaminated area of a site will be decontaminated. Various decontamination methods will be used to either physically remove contaminants, inactivate contaminants by disinfection or sterilization, or both. The physical decontamination techniques appropriate for equipment decontamination can be grouped into two categories: abrasive methods and non-abrasive methods.

Abrasive Cleaning Methods

Abrasive cleaning methods work by rubbing/scrubbing the surface containing the contaminant. This method includes mechanical and wet blasting methods.

Mechanical cleaning methods use brushes of metal or nylon. The amount and type of contaminants removed will vary with the hardness of bristles, length of brushing time, and degree of brush contact.

Cleaning can also be accomplished by water blasting which is also referred to as steam cleaning and pressure washing. Pressure washing utilizes high-pressure that is sprayed from a nozzle onto sampling equipment to physically remove soil or (potentially) contaminated material. Steam cleaning is a modification of pressure washing where the water is heated to temperatures approaching 100°C to assist in removing organic constituents from equipment.

Disinfection/Rinse Methods

Disinfectants are a practical means of inactivating chemicals or contaminants of concern. Standard sterilization methods involve heating the equipment which is impractical for large equipment. Rinsing removes contaminants through dilution, physical attraction, and solubilization.

The use of distilled/deionized water commonly available from commercial vendors may be acceptable for decontamination of sampling equipment provided that it has been verified by laboratory analysis to be target analyte free. Tap water may be used from any municipal water treatment system for mixing of decontamination solutions. An untreated potable water supply is not an acceptable substitute for tap water. Acids and solvents are occasionally utilized in decontamination of equipment to remove metals and organics, respectively, from sampling equipment. Other than ethanol, these are avoided when possible due to the safety, disposal, and transportation concerns associated with them.

Equipment or apparatuses that may be selected for use include the following:

- Personal protective clothing
- Non-phosphate detergent
- Selected solvents for removal of polar and nonpolar organics (ethanol, methanol, hexane)
- Acid washes for removal of metals (nitric acid)
- Long-handled brushes
- Drop cloths or plastic sheeting
- Paper towels
- Galvanized tubs or buckets
- Distilled, deionized, or tap water (as required by the project)
- Storage containers for spent wash solutions
- Sprayers (pressurized and non-pressurized)
- Trash bags
- Safety glasses or splash shield

<u>Field Sampling Equipment Cleaning Procedures</u>

The following procedures should be followed:

1. Where applicable, follow physical removal procedures previously described (pressure wash, scrub wash)
2. Wash equipment with a non-phosphate detergent solution
3. Rinse with tap water
4. Rinse with distilled or deionized water
5. Rinse with 10% nitric acid if the sample will be analyzed for metals/organics
6. Rinse with distilled or deionized water
7. Use a solvent rinse (pesticide grade) if the sample will be analyzed for organics
8. Air dry the equipment completely
9. Rinse again with distilled or deionized water

10. Place in clean bag or container for storage/transport to subsequent sampling locations.

Selection of the solvent for use in the decontamination process is based on the contaminants present at the site. Solvent rinses are not necessarily required when organics are not a contaminant of concern and may be eliminated from the sequence specified below. Similarly, an acid rinse is not required if the analyses do not include inorganics. Use of a solvent is required when organic contamination is present on-site. Typical solvents used for removal of organic contaminants include acetone, ethanol, hexane, methanol, or water. An acid rinse step is required if metals are present on-site. If a particular contaminant fraction is not present at the site, the ten-step decontamination procedure listed above may be modified for site specificity.

Sampling equipment that requires the use of plastic tubing should be disassembled and the tubing replaced with clean tubing before commencement of sampling and between sampling locations. Plastic tubing should not be reused.

INTRODUCTION

The following Standard Operating Procedure (SOP) presents the procedures for documenting activities observed or completed in the field in a field logbook. The documentation should represent all activities of WESTON personnel and entities under WESTON's supervision.

TERMS

FSP - Field Sampling Plan

SAP - Sampling and Analysis Plan

QAPP - Quality Assurance Project Plan

HASP - Health and Safety Plan

PROCEDURE

Field logbooks will be used and maintained during field activities to document pertinent information observed or completed by WESTON personnel or entities that WESTON is responsible for providing oversight. Field logbooks are legal documents that form the basis for later written reports and may serve as evidence in legal proceedings. The Site Manager or Field Team Leader will review field log entries daily and initial each page of entries. Field logbooks will be maintained by the Site Manager or Field Team Leader during field activities and transferred to the project files for a record of activities at the conclusion of the project. General logbook entry procedures are listed below.

- Logbooks must be permanently bound with all pages numbered to the end of the book. Entries should begin on page 1.
- Only use blue or black ink (waterproof) for logbook entries.
- Sign entries at the end of the day, or before someone else writes in the logbook.
- If a complete page is not used, draw a line diagonally across the blank portion of the page and initial and date the bottom line.
- If a line on the page is not completely filled, draw a horizontal line through the blank portion.
- Ensure that the logbook clearly shows the sequence of the day's events.
- Do not write in the margins or between written lines, and do not leave blank pages to fill in later.
- If an error is made, make corrections by drawing a single line through the error and initialing it.
- Maintain control of the logbook and keep in a secure location.

Field logbooks will contain, at a minimum, the following information, if applicable:

General Information

- Name, location of site, and work order number
- Name of the Site Manager or Field Team Leader
- Names and responsibilities of all field team members using the logbook (or involved with activities for which entries are being made)
- Weather conditions
- Field observations
- Names of any site visitors including entities that they represent

Sample Collection Activities

- Date(s) and times of the sample collection or event.
- Number and types of collected samples.
- Sample location with an emphasis on any changes to documentation in governing documents (i.e., SAP, FSP). This may include measurements from reference points or sketches of sample locations with respect to local features.
- Sample identification numbers, including any applicable cross-references to split samples or samples collected by another entity.
- A description of sampling methodology, or reference to any governing document (i.e., FSP, SAP, QAPP).
- Summary of equipment preparation and decontamination procedures.
- Sample description including depth, color, texture, moisture content, and evidence of waste material or staining.
- Air monitoring (field screening) results.
- Types of laboratory analyses requested.

Site Health and Safety Activities

- All safety, accident, and/or incident reports.

- Real-time personnel air monitoring results, if applicable, or if not documented in the HASP.
- Heat/cold stress monitoring data, if applicable.
- Reasons for upgrades or downgrades in personal protective equipment.
- Health and safety inspections, checklists (drilling safety guide), meetings/briefings.
- Calibration records for field instruments.

Oversight Activities

- Progress and activities performed by contractors including operating times.
- Deviations of contractor activities with respect to project governing documents (i.e., specifications).
- Contractor sampling results and disposition of contingent soil materials/stockpiles.
- Excavation specifications and locations of contractor confirmation samples.
- General site housekeeping and safety issues by site contractors.

SURFACE WATER SAMPLING

CONTENTS

CONTENTS (cont)

* These sections affected by Revision 0.0.

SUPERSEDES: SOP #2013; Revision 0.0; 11/17/94; U.S. EPA Contract EP-W-09-031.

SURFACE WATER SAMPLING

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) is applicable to the collection of representative surface water samples from streams, rivers, lakes, ponds, lagoons, and surface impoundments. It includes samples collected from depth, as well as samples collected from the surface.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute United States Environmental Protection Agency (U.S. EPA) endorsement or recommendation for use.

2.0 METHOD SUMMARY

Sampling situations vary widely; therefore, no universal sampling procedure can be recommended. However, surface water sampling is generally accomplished through the use of one of the following samplers or techniques:

- Kemmerer bottle
- Van Doren sampler
- Bacon bomb sampler
- Dip sampler
- Direct method

These samplers and sampling techniques will result in the collection of representative samples from the majority of surface waters and impoundments encountered.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Once samples have been collected, the following procedures should be followed:

1. Transfer the sample(s) into suitable, labeled sample containers specific for the analyses to be performed.

2. Preserve the sample, if appropriate, or use pre-preserved sample bottles. Do not overfill bottles if they are pre-preserved.

3. Cap the container securely, place in a resealable plastic bag, and cool to 4°C.

4. Record all pertinent data in the site logbook and/or on field data sheets.

5. Complete the Chain of Custody record.

6. Attach custody seals to cooler prior to shipment.

7. Decontaminate all non-dedicated sampling equipment prior to the collection of additional samples.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

There are two primary interferences or potential problems associated with surface water sampling. These include cross contamination of samples and improper sample collection.

1. Cross contamination problems can be eliminated or minimized through the use of dedicated or disposable sampling equipment. If this is not possible or practical, then decontamination of sampling equipment is necessary. Refer to ERT/SERAS SOP #2006, *Sampling Equipment Decontamination*.

2. Improper sample collection can involve using contaminated equipment, disturbance of the stream or impoundment substrate, and sampling in an obviously disturbed or non-representative area.

Following proper decontamination procedures, minimizing disturbance of the sample site, and careful selection of sampling locations will eliminate these problems. Proper timing for the collection of samples must be taken into consideration due to tidal influences and low or fast-flowing streams or rivers.

5.0 EQUIPMENT/APPARATUS

Equipment needed for collection of surface water samples may include (depending on technique chosen):

- Kemmerer bottles
- Van Doren sampler
- Bacon bomb sampler
- Dip sampler
- Line and messengers
- Peristalic pump
- Tygon tubing
- 0.45 micron (µm) filters
- Sample bottles/preservatives
- pH paper
- Resealable plastic bags
- Ice
- Coolers, packing material
- Chain of Custody records, custody seals
- Field data sheets
- Decontamination equipment/supplies
- Maps/plot plan
- Safety equipment
- Compass
- Tape measure
- Survey stakes, flags, or buoys and anchors
- Camera and film

- Logbook/waterproof pen
- Sample bottle labels
- Paper towels
- Disposable pipets
- Hydrolab

6.0 REAGENTS

Reagents will be utilized for preservation of samples and for decontamination of sampling equipment. The preservatives required are specified by the analysis to be performed and are summarized in ERT/SERAS SOP #2003, *Sample Storage, Preservation and Handling*. Decontamination solutions are specified in ERT/SERAS SOP #2006, *Sampling Equipment Decontamination.*

7.0 PROCEDURES

7.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.

2. Obtain the necessary sampling and monitoring equipment.

3. Decontaminate or pre-clean equipment, and ensure that it is in working order.

4. Prepare scheduling and coordinate with staff, clients, and regulatory agency, if appropriate.

5. Perform a general site survey prior to site entry, in accordance with the site specific Health and Safety Plan (HASP).

6. Use stakes, flags, or buoys to identify and mark all sampling locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and obstructions.

7.2 Representative Sampling Considerations

In order to collect a representative sample, the hydrology and morphometrics of a stream, river, pond, lake or impoundment should be determined prior to sampling. This will aid in determining the presence of phases or layers in lagoons or impoundments, flow patterns in streams, and appropriate sample locations and depths.

Water quality data should be collected in ponds, lakes and impoundments to determine if stratification is present. Measurements of dissolved oxygen, pH, conductivity, oxidation-potential, temperature and turbidity can indicate if strata exist that would affect analytical results. Measurements should be collected at one-meter intervals from the surface to the bottom using the appropriate instrument (i.e., a Hydrolab or equivalent). These water quality measurements can assist in the interpretation of analytical data, and the selection of sampling sites and depths when

surface water samples are collected.

Factors that contribute to the selection of a sampling device used for sampling surface waters in streams, rivers, lakes, ponds, lagoons, and surface impoundments are:

- Width, depth, flow and accessibility of the location being sampled
- Whether the sample will be collected onshore or offshore

7.2.1 Sampler Composition

The appropriate sampling device must be of a proper composition. Selection of samplers constructed of glass, stainless steel, polyvinyl chloride (PVC) or PFTE (Teflon) should be based upon the suspected contaminants and the analyses to be performed.

7.3 Sample Collection

7.3.1 Kemmerer Bottle

A Kemmerer bottle (Figure 1, Appendix A) may be used in most situations where site access is from a boat or structure, such as a bridge or pier, and where samples at specific depths are required. Sampling procedures are as follows:

1. Use a properly decontaminated Kemmerer bottle. Set the sampling device so that the upper and lower stoppers are pulled away from the body, allowing the surface water to enter tube.

2. Lower the pre-set sampling device to the predetermined depth. Avoid disturbance of the bottom.

3. When the Kemmerer bottle is at the required depth, send the weighted messenger down the suspension line, closing the sampling device.

4. Retrieve the sampler and discharge the first 10-20 milliliters (mL) from the drain to clear potential contamination from the valve. This procedure may be repeated if additional sample volume is needed to fulfill analytical requirements. Subsequent grabs may be composited or transferred directly to appropriate sample containers.

7.3.2 Van Doren Sampler

A Van Doren sampler (Figure 2, Appendix A) is used to collect surface water from a very specific sampling depth or from a shallow water body. Since the sampler is suspended horizontally, the depth interval sampled is the diameter of the sampling tube. The sampling procedure is as follows:

1. Use a properly decontaminated Van Doren sampler. Set the device so that the end stoppers are pulled away from the body allowing surface water to enter the

tube.

2. Lower the pre-set sampling device to the predetermined depth. Avoid disturbance of the bottom.

3. When the Van Doren is at the required depth, send the weighted messenger down the suspension line, closing the sampling device.

4. Retrieve the sampler and discharge the first 10-20 milliliters (mL) from the drain to clear potential contamination from the valve. This procedure may be repeated if additional sample volume is needed to fulfill analytical requirements. Subsequent grabs may be composited or transferred directly to appropriate sample containers.

7.3.3 Bacon Bomb Sampler

A bacon bomb sampler (Figure 3, Appendix A) may be used in situations similar to those outlined for the Kemmerer bottle. Sampling procedures are as follows:

1. Lower the bacon bomb sampler carefully to the desired depth, allowing the line for the trigger to remain slack at all times. When the desired depth is reached, pull the trigger line until taut. This will allow the sampler to fill.

2. Release the trigger line and retrieve the sampler.

3. Discharge the first 10-20 milliliters (mL) from the drain to clear potential contamination from the valve. This procedure may be repeated if additional sample volume is needed to fulfill analytical requirements. Subsequent grabs may be composited or transferred directly to appropriate sample containers.

7.3.4 Dip Sampler

A dip sampler (Figure 4, Appendix A) is useful in situations where a sample is to be recovered from an outfall pipe or along a lagoon bank where direct access is limited. The long handle on such a device allows access from a discrete location. Sampling procedures are as follows:

1. Assemble the device in accordance with the manufacturer's instructions.

2. Extend the device to the sample location and collect the sample by dipping the sampler into the water.

3. Retrieve the sampler and transfer the sample to the appropriate sample container(s).

7.3.5 Direct Method

For streams, rivers, lakes, and other surface waters, the direct method may be utilized to collect water samples directly into the sample container(s). Health and safety considerations must be addressed when sampling lagoons or other impoundments where specific conditions may exist that warrant the use of additional safety equipment. These issues must be addressed in the site-specific HASP.

Using adequate protective clothing, access the sampling station by appropriate means. For shallow stream stations, collect the sample under the water surface while pointing the sample container upstream; the container must be upstream of the collector. Avoid disturbing the substrate. For lakes and other impoundments, collect the sample under the water surface while avoiding surface debris and the boat wake.

When using the direct method, do not use pre-preserved sample bottles as the collection method may dilute the concentration of preservative necessary for proper sample preservation.

8.0 CALCULATIONS

This section is not applicable to this SOP.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

There are no specific quality assurance (QA) activities which apply to the implementation of these procedures. However, the following general QA procedures apply:

1. All data must be documented on field data sheets or within site logbooks.

2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation and they must be documented.

3. To avoid the incidental inclusion of disturbed sediment in the sample, surface water should be collected from a downstream to upstream direction and upstream of any activity that may disturb the sediment (i.e., wading).

4. While collecting surface water using the direct method, the sample container should be held below the surface to avoid the collection of floating debris.

5. Water quality data should be collected to detect the presence of stratified layers or other site-specific characteristics that would affect the sample.

10.0 DATA VALIDATION

This section is not applicable to this SOP.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, Occupational Health and Safety (OSHA) and corporate health and safety procedures.

More specifically, when sampling lagoons or surface impoundments containing known or suspected hazardous substances, adequate health and safety and boating precautions must be taken to ensure the safety of sampling personnel.

12.0 REFERENCES

Wilde, F.D., D.B. Radtke, J. Gibs and R.T. Iwatsubo. 1998. National Field Manual for the Collection of Water-Quality Data - Selection of Equipment for Water Sampling. U.S. Geological Survey Techniques of Water - Resources Investigations, Book 9, Chap. A2, variously paged.

http://water.usgs.gov/owq/FieldManual/index.htmland
http://water.usgs.gov/owq/FieldManual/mastererrat.html

U.S. Environmental Protection Agency. 1984. Characterization of Hazardous Waste Sites - A Methods Manual: Volume II. Available Sampling Methods, Second Edition. EPA/600/4-84-076.

13.0 APPENDICES

A - Figures

APPENDIX A
Figures
SOP #2013
February 2002

FIGURE 1. Kemmerer Bottle

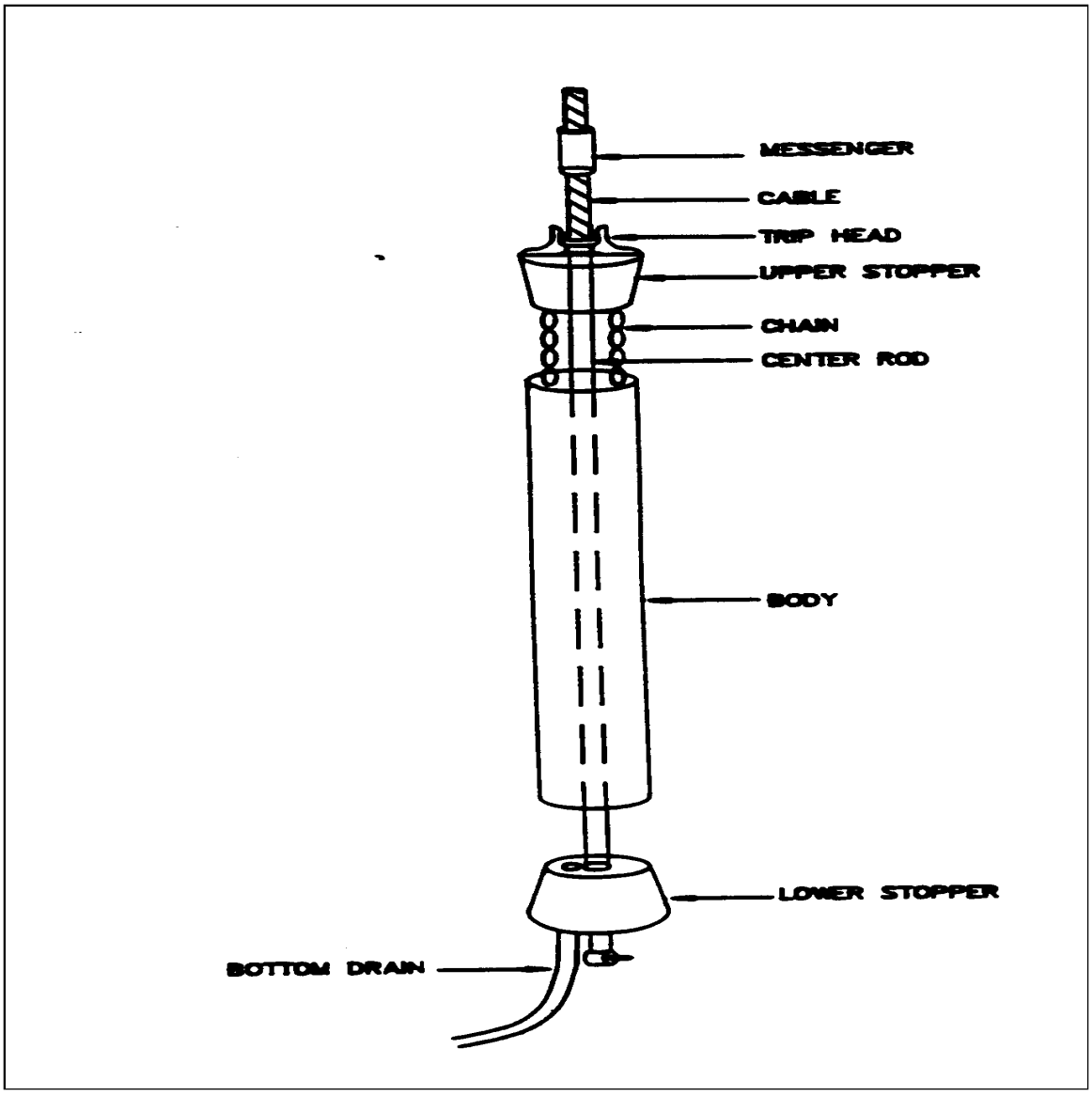


FIGURE 2. Van Doren Sampler

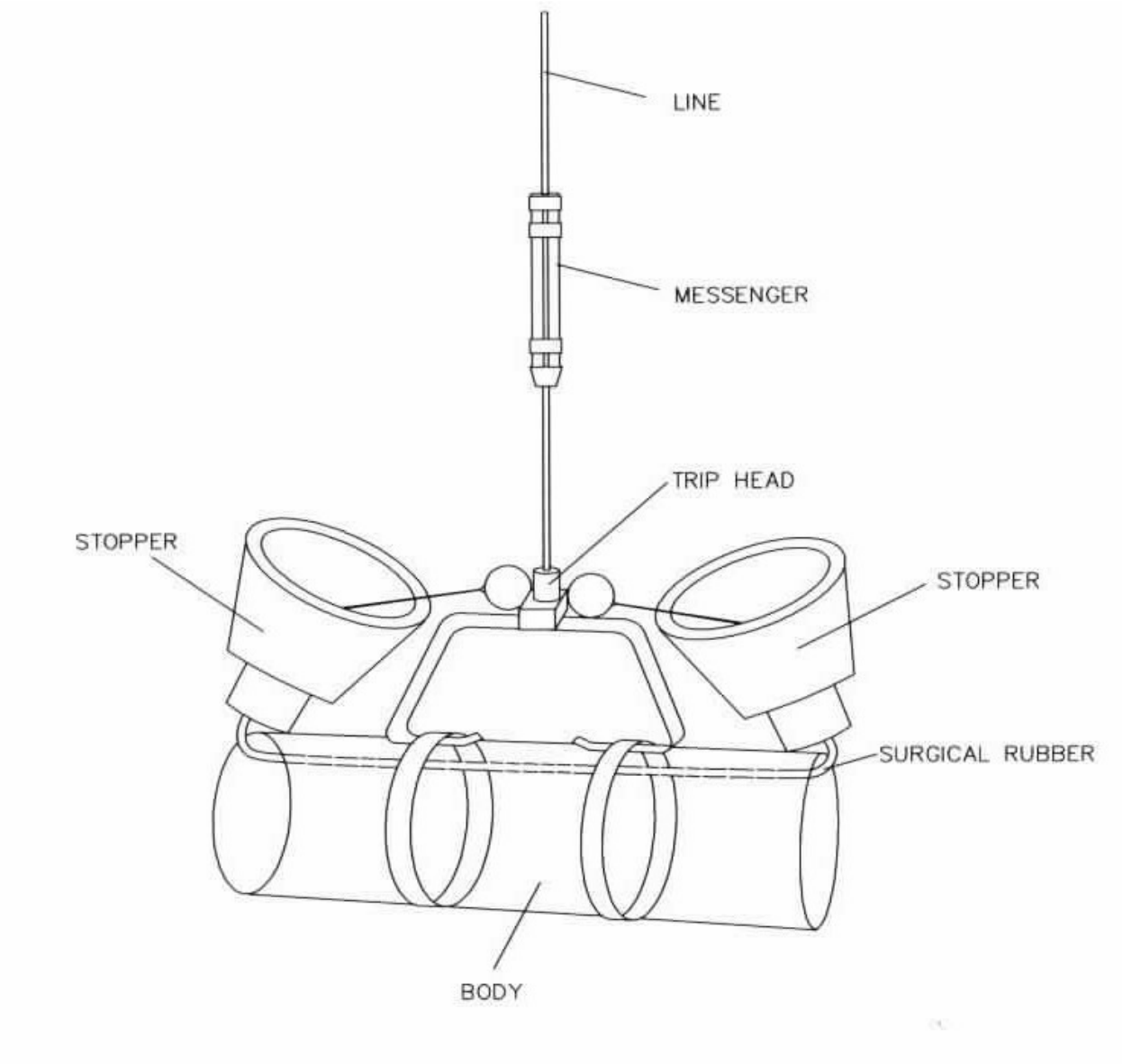


FIGURE 3. Bacon Bomb Sampler

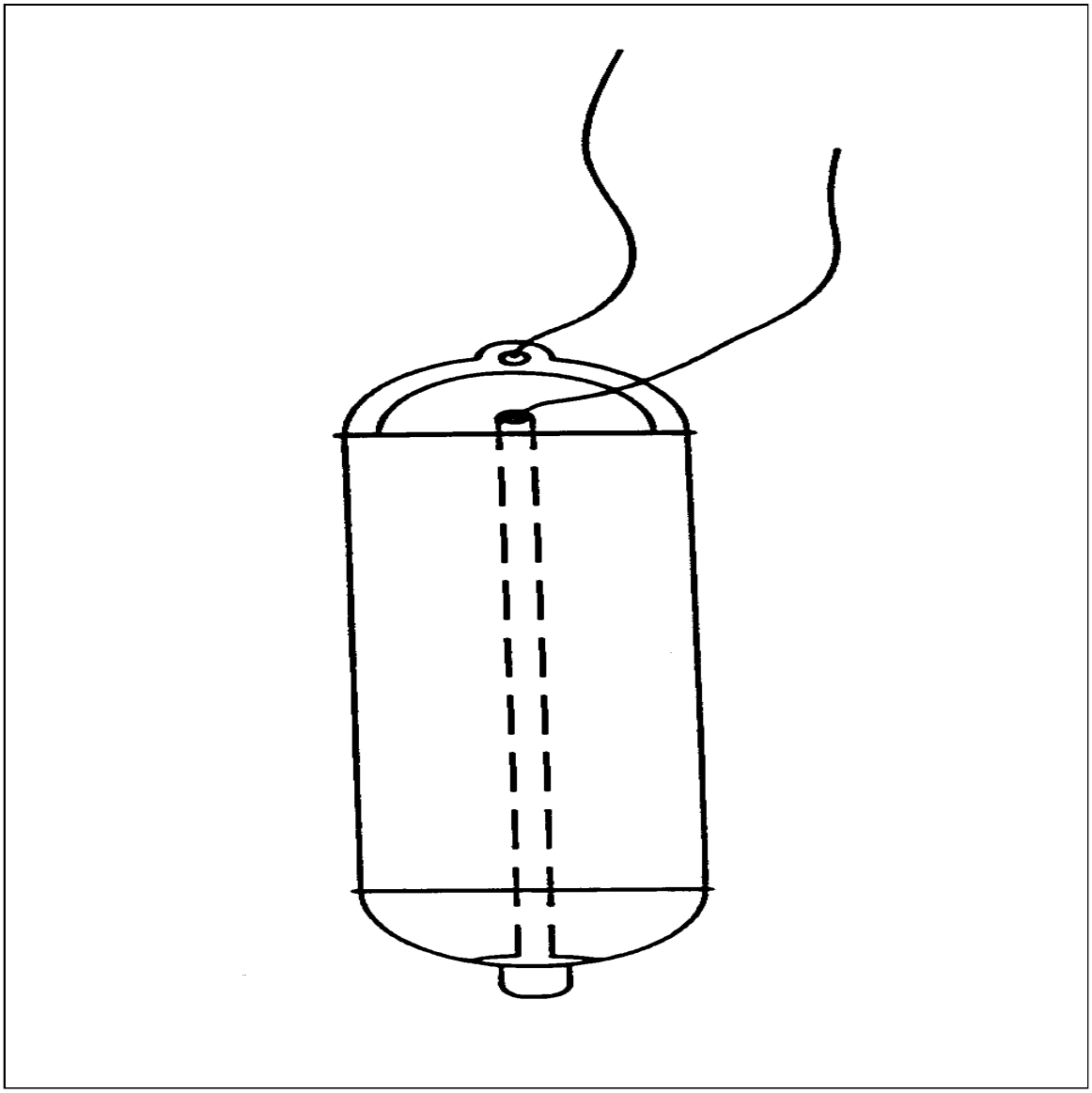
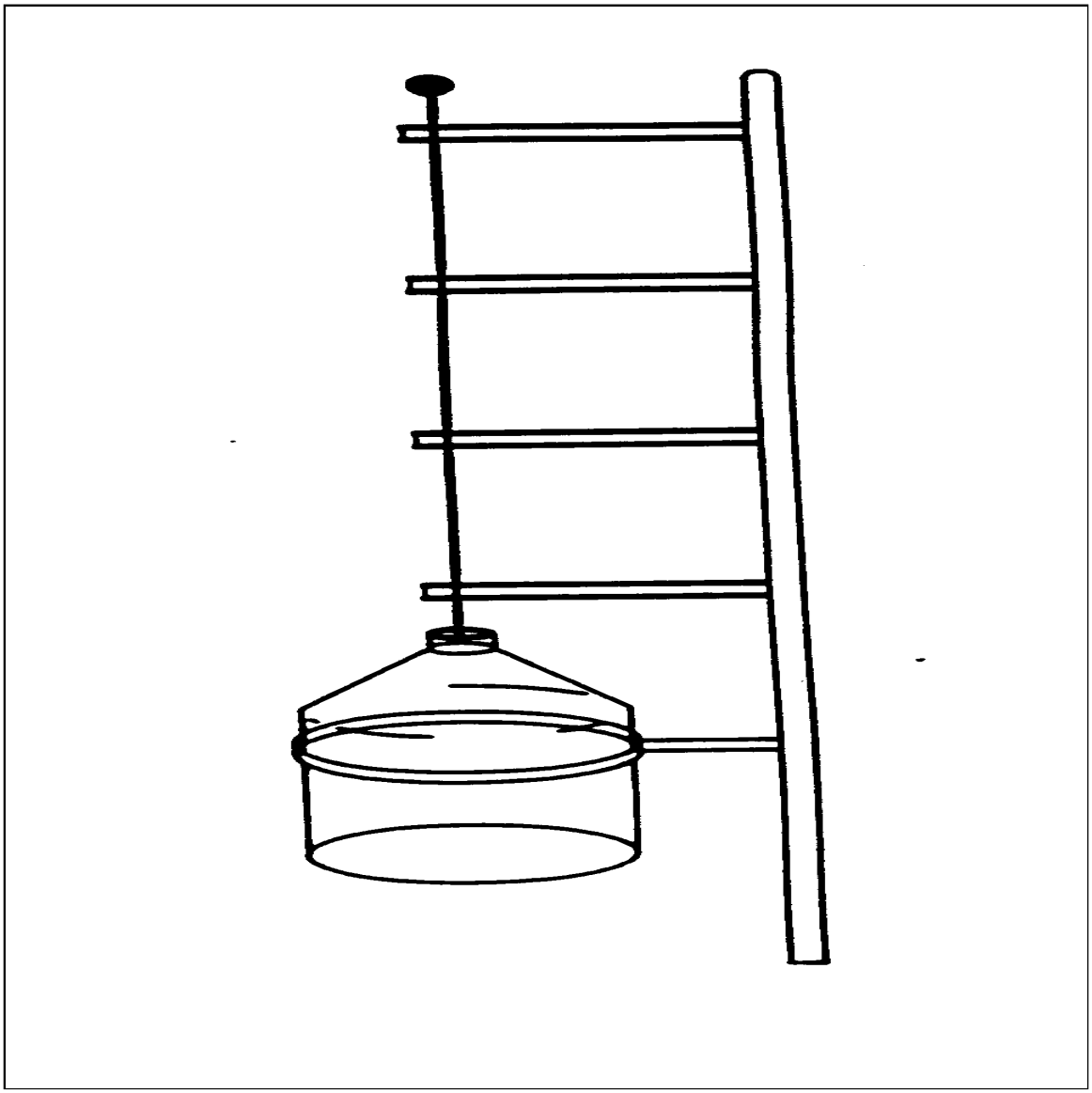


FIGURE 4. Dip Sampler



SAMPLING EQUIPMENT DECONTAMINATION

SOP#: 2006
DATE: 08/11/94
REV. #: 0.0

1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to provide a description of the methods used for preventing, minimizing, or limiting cross-contamination of samples due to inappropriate or inadequate equipment decontamination and to provide general guidelines for developing decontamination procedures for sampling equipment to be used during hazardous waste operations as per 29 Code of Federal Regulations (CFR) 1910.120. This SOP does not address personnel decontamination.

These are standard (i.e. typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitation, or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. Environmental Protection Agency (U.S. EPA) endorsement or recommendation for use.

2.0 METHOD SUMMARY

Removing or neutralizing contaminants from equipment minimizes the likelihood of sample cross contamination, reduces or eliminates transfer of contaminants to clean areas, and prevents the mixing of incompatible substances.

Gross contamination can be removed by physical decontamination procedures. These abrasive and non-abrasive methods include the use of brushes, air and wet blasting, and high and low pressure water cleaning.

The first step, a soap and water wash, removes all visible particulate matter and residual oils and grease. This may be preceded by a steam or high pressure water wash to facilitate residuals removal. The second step involves a tap water rinse and a distilled/deionized water rinse to remove the detergent. An acid rinse provides a low pH media for trace metals removal and is included in the decontamination process if metal samples are to be collected. It is followed by another distilled/deionized water rinse. If sample analysis does not include metals, the acid rinse step can be omitted. Next, a high purity solvent rinse is performed for trace organics removal if organics are a concern at the site. Typical solvents used for removal of organic contaminants include acetone, hexane, or water. Acetone is typically chosen because it is an excellent solvent, miscible in water, and not a target analyte on the Priority Pollutant List. If acetone is known to be a contaminant of concern at a given site or if Target Compound List analysis (which includes acetone) is to be performed, another solvent may be substituted. The solvent must be allowed to evaporate completely and then a final distilled/deionized water rinse is performed. This rinse removes any residual traces of the solvent.

The decontamination procedure described above may be summarized as follows:

1. Physical removal
2. Non-phosphate detergent wash
3. Tap water rinse
4. Distilled/deionized water rinse
5. 10% nitric acid rinse
6. Distilled/deionized water rinse
7. Solvent rinse (pesticide grade)
8. Air dry
9. Distilled/deionized water rinse

If a particular contaminant fraction is not present at the site, the nine (9) step decontamination procedure specified above may be modified for site specificity. For example, the nitric acid rinse may be eliminated if metals are not of concern at a site. Similarly, the solvent rinse may be eliminated if organics are not of

concern at a site. Modifications to the standard procedure should be documented in the site specific work plan or subsequent report.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

The amount of sample to be collected and the proper sample container type (i.e., glass, plastic), chemical preservation, and storage requirements are dependent on the matrix being sampled and the parameter(s) of interest.

More specifically, sample collection and analysis of decontamination waste may be required before beginning proper disposal of decontamination liquids and solids generated at a site. This should be determined prior to initiation of site activities.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

- The use of distilled/deionized water commonly available from commercial vendors may be acceptable for decontamination of sampling equipment provided that it has been verified by laboratory analysis to be analyte free (specifically for the contaminants of concern).

- The use of an untreated potable water supply is not an acceptable substitute for tap water. Tap water may be used from any municipal or industrial water treatment system.

- If acids or solvents are utilized in decontamination they raise health and safety, and waste disposal concerns.

- Damage can be incurred by acid and solvent washing of complex and sophisticated sampling equipment.

5.0 EQUIPMENT/APPARATUS

Decontamination equipment, materials, and supplies are generally selected based on availability. Other considerations include the ease of decontaminating or disposing of the equipment. Most equipment and supplies can be easily procured. For example, soft-bristle scrub brushes or long-handled bottle brushes can be used to remove contaminants. Large galvanized wash tubs, stock tanks, or buckets can hold wash and rinse solutions. Children's wading pools can also be used. Large plastic garbage cans or other similar containers lined with plastic bags can help segregate contaminated equipment. Contaminated liquid can be stored temporarily in metal or plastic cans or drums.

The following standard materials and equipment are recommended for decontamination activities:

5.1 Decontamination Solutions

- Non-phosphate detergent
- Selected solvents (acetone, hexane, nitric acid, etc.)
- Tap water
- Distilled or deionized water

5.2 Decontamination Tools/Supplies

- Long and short handled brushes
- Bottle brushes
- Drop cloth/plastic sheeting
- Paper towels
- Plastic or galvanized tubs or buckets
- Pressurized sprayers (H_2O)
- Solvent sprayers
- Aluminum foil

5.3 Health and Safety Equipment

Appropriate personal protective equipment (i.e., safety glasses or splash shield, appropriate gloves, aprons or coveralls, respirator, emergency eye wash)

5.4 Waste Disposal

- Trash bags
- Trash containers
- 55-gallon drums
- Metal/plastic buckets/containers for storage and disposal of decontamination solutions

6.0 REAGENTS

There are no reagents used in this procedure aside from the actual decontamination solutions. Table 1 (Appendix A) lists solvent rinses which may be required for elimination of particular chemicals. In

general, the following solvents are typically utilized for decontamination purposes:

- 10% nitric acid is typically used for inorganic compounds such as metals. An acid rinse may not be required if inorganics are not a contaminant of concern.
- Acetone (pesticide grade)[(1)]
- Hexane (pesticide grade)[(1)]
- Methanol[(1)]

[(1)] - Only if sample is to be analyzed for organics.

7.0 PROCEDURES

As part of the health and safety plan, a decontamination plan should be developed and reviewed. The decontamination line should be set up before any personnel or equipment enter the areas of potential exposure. The equipment decontamination plan should include:

- The number, location, and layout of decontamination stations.
- Decontamination equipment needed.
- Appropriate decontamination methods.
- Methods for disposal of contaminated clothing, equipment, and solutions.
- Procedures can be established to minimize the potential for contamination. This may include: (1) work practices that minimize contact with potential contaminants; (2) using remote sampling techniques; (3) covering monitoring and sampling equipment with plastic, aluminum foil, or other protective material; (4) watering down dusty areas; (5) avoiding laying down equipment in areas of obvious contamination; and (6) use of disposable sampling equipment.

7.1 Decontamination Methods

All samples and equipment leaving the contaminated area of a site must be decontaminated to remove any contamination that may have adhered to equipment. Various decontamination methods will remove contaminants by: (1) flushing or other physical action, or (2) chemical complexing to inactivate contaminants by neutralization, chemical reaction, disinfection, or sterilization.

Physical decontamination techniques can be grouped into two categories: abrasive methods and non-abrasive methods, as follows:

7.1.1 Abrasive Cleaning Methods

Abrasive cleaning methods work by rubbing and wearing away the top layer of the surface containing the contaminant. The mechanical abrasive cleaning methods are most commonly used at hazardous waste sites. The following abrasive methods are available:

Mechanical

Mechanical methods of decontamination include using metal or nylon brushes. The amount and type of contaminants removed will vary with the hardness of bristles, length of time brushed, degree of brush contact, degree of contamination, nature of the surface being cleaned, and degree of contaminant adherence to the surface.

Air Blasting

Air blasting equipment uses compressed air to force abrasive material through a nozzle at high velocities. The distance between nozzle and surface cleaned, air pressure, time of application, and angle at which the abrasive strikes the surface will dictate cleaning efficiency. Disadvantages of this method are the inability to control the amount of material removed and the large amount of waste generated.

Wet Blasting

Wet blast cleaning involves use of a suspended fine abrasive. The abrasive/water mixture is delivered by compressed air to the contaminated area. By using a very fine abrasive, the amount of materials removed can be carefully controlled.

7.1.2 Non-Abrasive Cleaning Methods

Non-abrasive cleaning methods work by forcing the contaminant off a surface with pressure. In general, the equipment surface is not removed using non-abrasive methods.

Low-Pressure Water

This method consists of a container which is filled with water. The user pumps air out of the container to create a vacuum. A slender nozzle and hose allow the user to spray in hard-to-reach places.

High-Pressure Water

This method consists of a high-pressure pump, an operator controlled directional nozzle, and a high-pressure hose. Operating pressure usually ranges from 340 to 680 atmospheres (atm) and flow rates usually range from 20 to 140 liters per minute.

Ultra-High-Pressure Water

This system produces a water jet that is pressured from 1,000 to 4,000 atmospheres. This ultra-high-pressure spray can remove tightly-adhered surface films. The water velocity ranges from 500 meters/second (m/s) (1,000 atm) to 900 m/s (4,000 atm). Additives can be used to enhance the cleaning action.

Rinsing

Contaminants are removed by rinsing through dilution, physical attraction, and solubilization.

Damp Cloth Removal

In some instances, due to sensitive, non-waterproof equipment or due to the unlikelihood of equipment being contaminated, it is not necessary to conduct an extensive decontamination procedure. For example, air sampling pumps hooked on a fence, placed on a drum, or wrapped in plastic bags are not likely to become heavily contaminated. A damp cloth should be used to wipe off contaminants which may have adhered to equipment through airborne contaminants or from surfaces upon which the equipment was set.

Disinfection/Sterilization

Disinfectants are a practical means of inactivating infectious agents. Unfortunately, standard sterilization methods are impractical for large equipment. This method of decontamination is typically performed off-site.

7.2 Field Sampling Equipment Decontamination Procedures

The decontamination line is setup so that the first station is used to clean the most contaminated item. It progresses to the last station where the least contaminated item is cleaned. The spread of contaminants is further reduced by separating each decontamination station by a minimum of three (3) feet. Ideally, the contamination should decrease as the equipment progresses from one station to another farther along in the line.

A site is typically divided up into the following boundaries: Hot Zone or Exclusion Zone (EZ), the Contamination Reduction Zone (CRZ), and the Support or Safe Zone (SZ). The decontamination line should be setup in the Contamination Reduction Corridor (CRC) which is in the CRZ. Figure 1 (Appendix B) shows a typical contaminant reduction zone layout. The CRC controls access into and out of the exclusion zone and confines decontamination activities to a limited area. The CRC boundaries should be conspicuously marked. The far end is the hotline, the boundary between the exclusion zone and the contamination reduction zone. The size of the decontamination corridor depends on the number of stations in the decontamination process, overall dimensions of the work zones, and amount of space available at the site. Whenever possible, it should be a straight line.

Anyone in the CRC should be wearing the level of protection designated for the decontamination crew. Another corridor may be required for the entry and exit of heavy equipment. Sampling and monitoring equipment and sampling supplies are all maintained outside of the CRC. Personnel don their equipment away from the CRC and enter the exclusion zone through a separate access control point at the hotline. One person (or more) dedicated to decontaminating equipment is recommended.

7.2.1 Decontamination Setup

Starting with the most contaminated station, the decontamination setup should be as follows:

Station 1: Segregate Equipment Drop

Place plastic sheeting on the ground (Figure 2, Appendix B). Size will depend on amount of

equipment to be decontaminated. Provide containers lined with plastic if equipment is to be segregated. Segregation may be required if sensitive equipment or mildly contaminated equipment is used at the same time as equipment which is likely to be heavily contaminated.

Station 2: Physical Removal With A High-Pressure Washer (Optional)

As indicated in 7.1.2, a high-pressure wash may be required for compounds which are difficult to remove by washing with brushes. The elevated temperature of the water from the high-pressure washers is excellent at removing greasy/oily compounds. High pressure washers require water and electricity.

A decontamination pad may be required for the high-pressure wash area. An example of a wash pad may consist of an approximately 1 1/2 foot-deep basin lined with plastic sheeting and sloped to a sump at one corner. A layer of sand can be placed over the plastic and the basin is filled with gravel or shell. The sump is also lined with visqueen and a barrel is placed in the hole to prevent collapse. A sump pump is used to remove the water from the sump for transfer into a drum.

Typically heavy machinery is decontaminated at the end of the day unless site sampling requires that the machinery be decontaminated frequently. A separate decontamination pad may be required for heavy equipment.

Station 3: Physical Removal With Brushes And A Wash Basin

Prior to setting up Station 3, place plastic sheeting on the ground to cover areas under Station 3 through Station 10.
Fill a wash basin, a large bucket, or child's swimming pool with non-phosphate detergent and tap water. Several bottle and bristle brushes to physically remove contamination should be dedicated to this station . Approximately 10 - 50 gallons of water may be required initially depending upon the amount of equipment to decontaminate and the amount of gross contamination.

Station 4: Water Basin

Fill a wash basin, a large bucket, or child's swimming pool with tap water. Several bottle and bristle brushes should be dedicated to this station. Approximately 10-50 gallons of water may be required initially depending upon the amount of equipment to decontaminate and the amount of gross contamination.

Station 5: Low-Pressure Sprayers

Fill a low-pressure sprayer with distilled/deionized water. Provide a 5-gallon bucket or basin to contain the water during the rinsing process. Approximately 10-20 gallons of water may be required initially depending upon the amount of equipment to decontaminate and the amount of gross contamination.

Station 6: Nitric Acid Sprayers

Fill a spray bottle with 10% nitric acid. An acid rinse may not be required if inorganics are not a contaminant of concern. The amount of acid will depend on the amount of equipment to be decontaminated. Provide a 5-gallon bucket or basin to collect acid during the rinsing process.

Station 7: Low-Pressure Sprayers

Fill a low-pressure sprayer with distilled/deionized water. Provide a 5-gallon bucket or basin to collect water during the rinsate process.

Station 8: Organic Solvent Sprayers

Fill a spray bottle with an organic solvent. After each solvent rinse, the equipment should be rinsed with distilled/deionized water and air dried. Amount of solvent will depend on the amount of equipment to decontaminate. Provide a 5-gallon bucket or basin to collect the solvent during the rinsing process.

Solvent rinses may not be required unless organics are a contaminant of concern, and may be eliminated from the station sequence.

Station 9: Low-Pressure Sprayers

Fill a low-pressure sprayer with distilled/deionized water. Provide a 5-gallon bucket or basin to collect water during the rinsate process.

Station 10: Clean Equipment Drop

Lay a clean piece of plastic sheeting over the bottom

plastic layer. This will allow easy removal of the plastic in the event that it becomes dirty. Provide aluminum foil, plastic, or other protective material to wrap clean equipment.

7.2.2 Decontamination Procedures

Station 1: Segregate Equipment Drop

Deposit equipment used on-site (i.e., tools, sampling devices and containers, monitoring instruments radios, clipboards, etc.) on the plastic drop cloth/sheet or in different containers with plastic liners. Each will be contaminated to a different degree. Segregation at the drop reduces the probability of cross contamination. Loose leaf sampling data sheets or maps can be placed in plastic zip lock bags if contamination is evident.

Station 2: Physical Removal With A High-Pressure Washer (Optional)

Use high pressure wash on grossly contaminated equipment. Do not use high- pressure wash on sensitive or non-waterproof equipment.

Station 3: Physical Removal With Brushes And A Wash Basin

Scrub equipment with soap and water using bottle and bristle brushes. Only sensitive equipment (i.e., radios, air monitoring and sampling equipment) which is waterproof should be washed. Equipment which is not waterproof should have plastic bags removed and wiped down with a damp cloth. Acids and organic rinses may also ruin sensitive equipment. Consult the manufacturers for recommended decontamination solutions.

Station 4: Equipment Rinse

Wash soap off of equipment with water by immersing the equipment in the water while brushing. Repeat as many times as necessary.

Station 5: Low-Pressure Rinse

Rinse sampling equipment with distilled/deionized water with a low-pressure sprayer.

Station 6: Nitric Acid Sprayers (required only if metals are a contaminant of concern)

Using a spray bottle rinse sampling equipment with nitric acid. Begin spraying (inside and outside) at one end of the equipment allowing the acid to drip to the other end into a 5-gallon bucket. A rinsate blank may be required at this station. Refer to Section 9.

Station 7: Low-Pressure Sprayers

Rinse sampling equipment with distilled/deionized water with a low-pressure sprayer.

Station 8: Organic Solvent Sprayers

Rinse sampling equipment with a solvent. Begin spraying (inside and outside) at one end of the equipment allowing the solvent to drip to the other end into a 5-gallon bucket. Allow the solvent to evaporate from the equipment before going to the next station. A QC rinsate sample may be required at this station.

Station 9: Low-Pressure Sprayers

Rinse sampling equipment with distilled/deionized water with a low-pressure washer.

Station 10 : Clean Equipment Drop

Lay clean equipment on plastic sheeting. Once air dried, wrap sampling equipment with aluminum foil, plastic, or other protective material.

7.2.3 Post Decontamination Procedures

1. Collect high-pressure pad and heavy equipment decontamination area liquid and waste and store in appropriate drum or container. A sump pump can aid in the collection process. Refer to the Department of Transportation (DOT) requirements for appropriate containers based on the contaminant of concern.

2. Collect high-pressure pad and heavy equipment decontamination area solid waste and store in appropriate drum or container. Refer to the DOT requirements for appropriate containers based on the contaminant of concern.

3. Empty soap and water liquid wastes from basins and buckets and store in appropriate

drum or container. Refer to the DOT requirements for appropriate containers based on the contaminant of concern.

4. Empty acid rinse waste and place in appropriate container or neutralize with a base and place in appropriate drum. pH paper or an equivalent pH test is required for neutralization. Consult DOT requirements for appropriate drum for acid rinse waste.

5. Empty solvent rinse sprayer and solvent waste into an appropriate container. Consult DOT requirements for appropriate drum for solvent rinse waste.

6. Using low-pressure sprayers, rinse basins, and brushes. Place liquid generated from this process into the wash water rinse container.

7. Empty low-pressure sprayer water onto the ground.

8. Place all solid waste materials generated from the decontamination area (i.e., gloves and plastic sheeting, etc.) in an approved DOT drum. Refer to the DOT requirements for appropriate containers based on the contaminant of concern.

9. Write appropriate labels for waste and make arrangements for disposal. Consult DOT regulations for the appropriate label for each drum generated from the decontamination process.

8.0 CALCULATIONS

This section is not applicable to this SOP.

9.0 QUALITYASSURANCE/ QUALITY CONTROL

A rinsate blank is one specific type of quality control sample associated with the field decontamination process. This sample will provide information on the effectiveness of the decontamination process employed in the field.

Rinsate blanks are samples obtained by running analyte free water over decontaminated sampling equipment to test for residual contamination. The blank water is collected in sample containers for handling, shipment, and analysis. These samples are treated identical to samples collected that day. A rinsate blank is used to assess cross contamination brought about by improper decontamination procedures. Where dedicated sampling equipment is not utilized, collect one rinsate blank per day per type of sampling device samples to meet QA2 and QA3 objectives.

If sampling equipment requires the use of plastic tubing it should be disposed of as contaminated and replaced with clean tubing before additional sampling occurs.

10.0 DATA VALIDATION

Results of quality control samples will be evaluated for contamination. This information will be utilized to qualify the environmental sample results in accordance with the project's data quality objectives.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow OSHA, U.S. EPA, corporate, and other applicable health and safety procedures.

Decontamination can pose hazards under certain circumstances. Hazardous substances may be incompatible with decontamination materials. For example, the decontamination solution may react with contaminants to produce heat, explosion, or toxic products. Also, vapors from decontamination solutions may pose a direct health hazard to workers by inhalation, contact, fire, or explosion.

The decontamination solutions must be determined to be acceptable before use. Decontamination materials may degrade protective clothing or equipment; some solvents can permeate protective clothing. If decontamination materials do pose a health hazard, measures should be taken to protect personnel or substitutions should be made to eliminate the hazard. The choice of respiratory protection based on contaminants of concern from the site may not be appropriate for solvents used in the decontamination process.

Safety considerations should be addressed when using abrasive and non-abrasive decontamination

equipment. Maximum air pressure produced by abrasive equipment could cause physical injury. Displaced material requires control mechanisms.

Material generated from decontamination activities requires proper handling, storage, and disposal. Personal Protective Equipment may be required for these activities.

Material safety data sheets are required for all decontamination solvents or solutions as required by the Hazard Communication Standard (i.e., acetone, alcohol, and trisodiumphosphate).

In some jurisdictions, phosphate containing detergents (i.e., TSP) are banned.

12.0 REFERENCES

Field Sampling Procedures Manual, New Jersey Department of Environmental Protection, February, 1988.

A Compendium of Superfund Field Operations Methods, EPA 540/p-87/001.

Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual, USEPA Region IV, April 1, 1986.

Guidelines for the Selection of Chemical Protective Clothing, Volume 1, Third Edition, American Conference of Governmental Industrial Hygienists, Inc., February, 1987.

Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities, NIOSH/OSHA/USCG/EPA, October, 1985.

APPENDIX A

Table

Table 1. Soluble Contaminants and Recommended Solvent Rinse

TABLE 1 Soluble Contaminants and Recommended Solvent Rinse		
SOLVENT[1]	EXAMPLES OF SOLVENTS	SOLUBLE CONTAMINANTS
Water	Deionized water Tap water	Low-chain hydrocarbons Inorganic compounds Salts Some organic acids and other polar compounds
Dilute Acids	Nitric acid Acetic acid Boric acid	Basic (caustic) compounds (e.g., amines and hydrazines)
Dilute Bases	Sodium bicarbonate (e.g., soap detergent)	Acidic compounds Phenol Thiols Some nitro and sulfonic compounds
Organic Solvents [2]	Alcohols Ethers Ketones Aromatics Straight chain alkalines (e.g., hexane) Common petroleum products (e.g., fuel, oil, kerosene)	Nonpolar compounds (e.g., some organic compounds)
Organic Solvent[2]	Hexane	PCBs

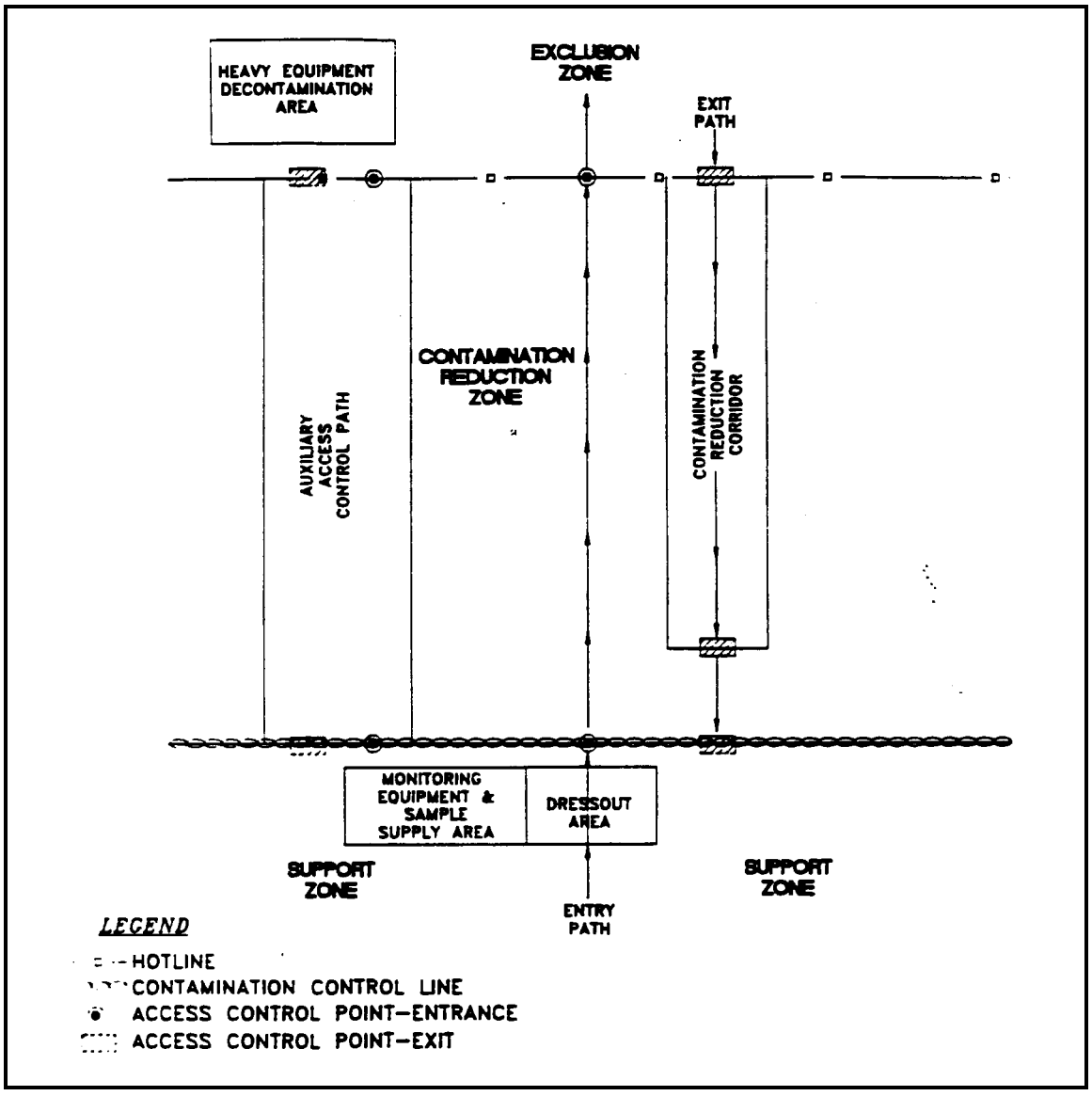
[1] - Material safety data sheets are required for all decontamination solvents or solutions as required by the Hazard Communication Standard

[2] - WARNING: Some organic solvents can permeate and/or degrade the protective clothing

APPENDIX B

Figures

Figure 1. Contamination Reduction Zone Layout



APPENDIX B (Cont'd.)

Figures

Figure 2. Decontamination Layout